


GENERAL RADIOCHEMISTRY AND ROUTINE ANALYTICAL SERVICES PROTOCOL (GRRASP)

PART B RADIOANALYTICAL SERVICES PROTOCOL (RASP)

STATEMENT OF WORK

 **EG&G ROCKY FLATS**
ENVIRONMENTAL MANAGEMENT DEPARTMENT
ROCKY FLATS PLANT
GOLDEN, COLORADO

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By Paul J. [signature]
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SCOPE OF WORK PART B

Radioanalytical Services for Rocky Flats Plant Environmental Management Department

B1.0 INTRODUCTION

This section of the Scope of Work pertains to the procurement of radioanalytical services for samples collected in support of the Rocky Flats Plant Environmental Management Department and is designated as Radiochemistry Analytical Services Protocol (RASAP). Radioanalytical services are needed for the parameters indicated below. The sample matrices may consist of air filters, biota, granular activated carbon (GAC), groundwater, mixed matrices, plastics, polycarbonate filters, sediment, sludge, soil, surface water, vegetation, waste filter socks, and other solids as appropriate for the methods.

1.1 General Radiochemistry Parameters

Gross Alpha and Beta
Tritium (by Liquid Scintillation)
Quantity: Up to 1000 samples

1.2 Selected Radiochemistry Isotopes

²⁴¹ Americium
¹³⁴ Cesium
¹³⁷ Cesium
²⁴⁴ Curium
²³⁷ Neptunium
²³⁸ Plutonium (for air filters)
²³⁹⁺²⁴⁰ Plutonium
²²⁶ Radium
²²⁸ Radium
⁸⁹⁺⁹⁰ Strontium
²³⁰⁺²³² Thorium
²³³⁺²³⁴ Uranium
²³⁵ Uranium
²³⁸ Uranium

Quantity: up to 1000 samples.

Bidders' technical proposals should provide adequate detail regarding the technical, analytical, evidentiary, documentary, and reporting approaches to be used. EG&G Rocky Flats (EG&G) anticipates that any and all data and documentation generated as a result of this procurement may be subjected to intense regulatory and public scrutiny; all data and documentation may also be discoverable in any resulting legal proceedings. For these reasons, bidders are requested to address how they intend to maintain and demonstrate in the laboratory the security and integrity of Rocky Flats Plant samples, extracts, data, and documentation.

B2.0 TECHNICAL REQUIREMENTS

The following are the technical requirements which comprise this procurement:

- 2.1 The subcontractor shall analyze up to 1000 samples comprised of any combination of the following matrices: Ground Water, Surface Water, Soils, Sludges, Sediments, Subsurface Soils, Biota, Granular Activated Carbon, Fabrics, Plastics, Waste Filter Socks, Polycarbonate Filters, and mixed-matrices. Up to 150 Pond Discharge samples will also be analyzed.
- 2.2 Samples shall be analyzed within 61 days after Validated Time of Sample Receipt (VTSR).
- 2.3 Water samples to be analyzed for dissolved radiochemistry parameters shall be filtered in the field. Total Radiochemistry water samples may be filtered in the laboratory when they contain sediment organic matter. The filters shall then be digested and added to the water aliquot prior to analysis. When required, filters may be analyzed separately as long as results are added to the water aliquot results and reported as total.
- 2.4 Total digestion/dissolution is required for soils, sediments and waste filter socks unless otherwise specified by EG&G.
- 2.5 The detection limits needed for the parameters listed in section 1.0 (above) are specified in Table B1 in ATTACHMENT I (REQUIREMENTS FOR SAMPLE ANALYSIS, DELIVERABLES, AND DOCUMENTATION SUPPLIED BY RADIOANALYTICAL LABORATORY SUBCONTRACTORS).
- 2.6 Analyses for radiochemistry parameters shall meet all the requirements specified in ATTACHMENT I, Exhibit I (RADIOCHEMISTRY ANALYTICAL REQUIREMENTS). Laboratories shall modify their standard radiochemistry methods to comply in all respects to the Exhibit I requirements. Any deviations must be approved and documented by EG&G prior to analysis. The requirements given were developed for water samples; however, these requirements also apply analogously to other matrices.
- 2.7 The laboratory shall make the decision to analyze Radium in water samples based upon the following decision scheme:

if Gross alpha is > 5.0 pCi/L, analyze for ^{226}Ra ;
if ^{226}Ra is > 3.0 pCi/L, analyze for ^{228}Ra .

For soil samples, both radiums shall be analyzed.

- 2.8 Deliverables include those specified in Table 3 (Radiochemistry Reports/Deliverables) of ATTACHMENT I and shall be submitted according to the schedule also included in Table 3. The format and content of data packages submitted for review and validation must conform in all respects to the requirements specified in ATTACHMENT I, Exhibit II (DATA PACKAGE CHECKLISTS). The contractor's proposal shall specifically include a detailed plan for ensuring the completeness and accuracy of data packages and deliverables.

- 2.9 The subcontractor shall submit two controlled copies of written Standard Operating Procedures (SOPs) including one controlled copy of the lab's Quality Assurance Program Plan for each laboratory facility to EG&G within 30 days of contract award or prior to receipt of samples, whichever comes first, and the SOPs shall conform to the requirements specified in Section III, part C of ATTACHMENT 1.

The subcontractor must at all times strictly adhere to the SOPs explicitly as written. Deviations made at the benchtop by any laboratory personnel must be approved by the laboratory's Quality Assurance Department and shall be documented in the Case Narratives submitted with each data package. If the deviation is to become a permanent change, two controlled copies of the revision of the laboratory's SOP shall be forwarded to the EG&G Radioanalytical Program Chemist.

EG&G understands that the subcontractor may consider their SOPs to be confidential documents. EG&G agrees that contractor SOP manuals and Quality Assurance/Quality Control (QA/QC) plan documents will be maintained in confidentiality and will not be copied or distributed outside EG&G without the consent and acknowledgment of the subcontractor.

- 2.10 The subcontractor shall be subject to routine, on-site technical audits and inspection by EG&G, (or designated representatives) not more than two times per calendar year during performance of this work. The preliminary pre-award technical audit performed prior to contract award does not count as one of these routine audits. Laboratories shall make available during audits all requested data and documentation related to this work. Results of audits are documented and corrective action responses for audit findings, including a schedule for implementation, are required.

On-site inspections, surveillances, or audits (announced or unannounced) for the purpose of identifying and resolving deficiencies or verifying corrective action may be performed by EG&G at any time during performance of the contract.

- 2.11 Laboratories shall be presently participating in a performance evaluation (PE) sample analysis program recognized by EPA or regulatory agencies, e.g. Drinking Water Certification. Laboratories shall score at least 70% on these PEs. Evidence of current participation or PE scores must be included in bidder's proposal.
- 2.12 Sample Holding Times are defined as the duration between date of sample collection and date of sample preparation (digestion/dissolution) and analysis. For water samples, the holding times shall be 180 days. For soil and other solid matrix samples, no holding times exists are specified, but 180 days shall be used as a guideline for these analyses.
- 2.13 The subcontractor shall submit Minimum Detectable Activities (MDAs), as appropriate, for each sample. Actual numerical values for analytical results shall be reported, rather than "< MDA."

2.14 The following guidance shall be used for developing Sample Delivery Groups (SDGs) or "batches":

- Surface waters and pond waters shall be grouped together for the purpose of batching.
- Groundwaters (well waters) shall be grouped together for batching.
- An EG&G "batch" shall follow the EPA CLP definition of an SDG. A batch is defined as 20 samples OR those samples received within 14 days inclusive.
- The Laboratories shall accumulate radiochemical samples "in-house" in groups of 20 to minimize the number of quality control samples being run. When necessary to meet holding times, smaller batches shall be analyzed.

2.15 Sample analyses shall be conducted using standard methods and shall meet the requirements specified in Exhibit I, Radiochemistry Analytical Requirements. Analyses shall be conducted under a documented QA/QC program. EG&G expects laboratories to adhere to standard, accepted QA/QC procedures and applicable *Good Laboratory Practice Standards* (40 CFR 792) during analysis of EG&G samples.

2.16 Results of analyses shall be reported using the units specified below:

<u>Matrix</u>	<u>Units</u>
Air Filters	µCi/ml
Biota	pCi/sample type
Granular Activated Carbon	pCi/g
Sediment	pCi/g
Sludge	pCi/g
Soil	pCi/g (dry)
Vegetation	pCi/g ashed
Waste Filter Socks	pCi/g of composite
Water	pCi/L

2.17 The subcontractors shall compile and submit quarterly histories of RFP blanks for alpha spectrometry analyses.

B3.0 ADDITIONAL INFORMATION FOR BID

The following items are to be considered for preparing bids:

- 3.1 Unit prices shall be quoted for all analyses including both Dissolved and Total Radiochemistry.
- 3.2 EG&G may request that technical representatives from the laboratories participating in this program attend periodic technical workshops. These workshops will be held in the Denver Metro Area and will typically last for 2 days. The subcontractor shall include in the proposal a unit price/meeting for attending these workshops.
- 3.3 The subcontractor shall provide bottles as required to be used for Radiochemistry samples. The proper type bottles shall be designated by the field subcontractors.

- 3.4 For radiochemistry analyses, the subcontractor is required to generate self-absorption curves at least every three years for calibration of detectors on alpha and beta proportional counting systems. Any reconfiguration, repair, or replacement of instrument components must also be accompanied by initial calibration of the system. Quality control samples are analyzed at a frequency of 5%, except replicate samples, which are analyzed at a frequency of 10%. The cost for analyzing the Laboratory Control Sample (LCS) shall be charged as a separate line item.

- 3.5 The subcontractor may use deionized water for preparing laboratory soil blanks.

B4.0 ACCEPTANCE OF DATA

Due to the stringent reporting requirements imposed at Rocky Flats Plant by various oversight and regulatory agencies, analytical data may need to be used prior to actual acceptance. Because of these requirements, for samples analyzed under this procurement, preliminary use of the data will not necessarily constitute acceptance.

EG&G and its designated representatives routinely review and validate analytical data according to EPA and internally developed functional guidelines. Validation reports are provided to the data users and to the laboratories that generated the data. Only complete and validatable data packages will be accepted. Incomplete, illegible, or unusable data packages will not be accepted. The subcontractor shall be expected to provide missing data within a specified time frame, and make all necessary corrections to the data packages to make them acceptable. Invoices for data which are "non-validatable" will not be approved for payment.

B5.0 SPECIAL TERMS AND CONDITIONS

- 3 The following special terms and conditions apply to this procurement:

- 5.1 Some samples may contain radioactivity at levels which exceed twice natural background of 36 mR/Hour ("background" levels for the general vicinity of the Plant are considered to be 18 mR/Hour). Background radiation is defined as the natural level of radioactivity indigenous in specific geographical areas due to geological origin of the material, topographics, and radiation from internally-deposited, naturally occurring radionuclides.

Because of this possibility, the subcontractor shall provide suitably licensed facilities to handle analyses of samples with elevated activity levels. All radiochemistry analyses shall be conducted in licensed facilities. Copies of a laboratory's NRC/State radioactive license shall be submitted with the subcontractor's proposal.

All samples are prescreened for activity levels prior to being shipped off Plant-site except terminal discharge pond samples and those samples which historically exhibit <50 pCi. EG&G shall apprise the subcontractor of any samples showing elevated activity levels.

- 5.2 Disposal of residual samples and extracts is the responsibility of the laboratory, unless it is determined that the residuals are not within USEPA permitted capabilities of the subcontractor or their designated vendor. Such samples may be returned to Rocky Flats Plant for proper disposition. It is anticipated that most samples will not meet the USEPA's criteria of "mixed-waste." By definition, a mixed-waste is radioactive ($>2\text{nCi}$) and contains organic compounds and/or hazardous/toxic compounds above Plant background. Those samples meeting the criteria of a mixed-waste are to be returned to Rocky Flats Plant. The subcontractor will be notified of any samples which may meet this criteria.

ATTACHMENT I

REQUIREMENTS FOR SAMPLE ANALYSIS, DELIVERABLES AND DOCUMENTATION SUPPLIED BY RADIOANALYTICAL LABORATORY SUBCONTRACTORS

REQUIREMENTS FOR SAMPLE ANALYSIS,
DELIVERABLES, AND DOCUMENTATION SUPPLIED
BY RADIOANALYTICAL LABORATORY SUBCONTRACTORS

1. GENERAL REQUIREMENTS

Sample analyses requirements for the Rocky Flats Plant (RFP) Environmental Management are consistent with those specified in Statements of Work (SOWs) used in the USEPA's Contract Laboratory Program (CLP).

1. *USEPA-CLP, 1987, Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration.*

The subcontractor may not deviate from these SOW requirements except as specified in this requirements document, or with written consent of the contracting officer.

For analyses where CLP methods are not available, such as radiochemistry (gross alpha/beta, $^{239/240}\text{Pu}$, $^{233/234,235\&238}\text{U}$, tritium, total radiostrontium, ^{137}Cs , etc.), the following methods apply:

1. American National Standards Institute (ANSI) N42.14, 1978, "American National Standard Calibration and Use of Germanium Detectors for the Measurement of Gamma Ray Emission Rates of Radionuclides."
2. ANSI N13.30 (draft), 1985, Health Physics Society Subcommittee WG 2-5, "Performance Criteria for Radiobioassay."
3. ANSI N42.15, 1980, "American National Standard Performance Verification of Liquid - Scintillation Counting Systems."
4. American Society for Testing Materials (ASTM) E 182-82, 1983, "Standard General Methods for Detector Calibration and Analysis of Radionuclides."
5. ASTM, 1979, *1979 Annual Book of ASTM Standards, Part 31*, Philadelphia, PA.
6. ASTM, 1978, *1978 Annual Book of ASTM Standards", Part 31*, Philadelphia, PA.
7. Kirchmer, C. J., 1983, *Quality Control in Water Analyses* in Environmental Science Technology, Vol. 17, No. 4.
8. USEPA, 1986, *Test Methods for Evaluating Solid Waste*, 3rd Edition, SW-846.
9. USEPA, 1979, *Radiochemical Analytical Procedures for Analysis of Environmental Samples*, No. EMSL-LV-0539-17, Las Vegas, Nevada.

10. USEPA, 1976, *Interim Radiochemical Methodology for Drinking Water*, No. EPA-600/4-75-008, Cincinnati, Ohio.
11. USEPA, 1980, *Prescribed Procedures for Measurement of Radioactivity in Drinking Water*, No. EPA-600/4-80-032, Cincinnati, Ohio.
12. American Public Health Association (APHA), 1985, *Standard Methods for the Examination of Water and Wastewater*, 16th Edition, New York, New York.
13. USEPA, 1984, *Eastern Environmental Radiation Facility Radiochemistry Procedures Manual*, No. 520/5-84-006.
14. USEPA, *Methods for Chemical Analysis of Water and Wastes*, No. 600/4-79-020, 1983.
15. U.S. Department of Energy, 1988, *The Environmental Survey Manual*, Appendix D, part 4 (Radiochemical Analysis Procedures), 2nd Edition.
16. USEPA, 1973, *Procedures for Radiochemical Analysis of Nuclear Reactor Aqueous Solutions*, No. R4-73-014.
17. Knoll, G. F., 1979, *Radiation Detection and Measurement*, 2nd Edition, Wiley and Sons, San Francisco, CA.
18. Harley, J.H., 1972, *HASL Procedures Manual*, HASL-300, U.S. Atomic Energy Commission, New York, New York.
19. *Good Laboratory Practice Standards*, 1989, (40 CFR 792).
20. NRC Regulatory Guides.

The methods for particular analyses must be used such that the Required Detection Limits (RDLs) [or Minimum Detectable Activities (MDAs)] are achieved. Standard methods may be modified or alternative methods substituted only with the written consent of the contracting officer. Specific MDA requirements for radiochemical analyses are described in Section II B, below (Tables 2 and 3).

II. ANALYTICAL REQUIREMENTS

A. Media Types

Environmental samples may consist of any of the following matrices:

- | | |
|------------------------------------|--------------------------------|
| 1. Air Filters | 10. Soil (and subsurface soil) |
| 2. Biota | 11. Surface Water |
| 3. Granular Activated Carbon (GAC) | 12. Vegetation |
| 4. Groundwater | 13. Waste Filter Socks |
| 5. Mixed Matrices | |
| 6. Plastics | |
| 7. Polycarbonate Filters | |
| 8. Sediment | |
| 9. Sludge | |

Aqueous samples may be filtered or non-filtered. For some studies, both filtered and non-filtered samples are analyzed.

B. Radiochemistry Parameters

Sample analyses shall be conducted using standard methods and shall meet the requirements specified in Exhibit I, Radiochemistry Analytical Requirements. Analyses shall be conducted under a documented quality assurance/quality control (QA/QC) program.

Samples may be analyzed for any or all of the parameters listed below. RDL's for these parameters are listed below.

TABLE 1				
Radiochemistry Parameters Required Detection Limit (RDL)				
Parameter	CAS No.	Water (pCi/L)	Soil (dry) (pCi/g)	Waste Filter Socks (pCi/g Composite)
Gross Alpha, dissolved	10-79-7	2	4	4
Gross Beta, dissolved	10-81-1	4	10	10
Gross Alpha, suspended	10-78-6	2	4	4
Gross Beta, suspended	10-80-0	4	10	10
Tritium	10028-17-8	400	400 (pCi/L)	
^{239/240} Pu	10-12-8	0.01	0.03	.07
^{233/234} U	11-08-5	0.6	0.3	0.10
²³⁵ U	15117-96-1	0.6	0.3	.05
²³⁸ U	7440-60-1	0.6	0.3	0.10
²⁴¹ Americium	14596-10-2	0.01	0.02	.07
Total Radiostrontium	11-10-9	1	1	
Total Radiocesium	13-00-0	1	0.1	
²²⁶ Radium	13982-63-3	0.5	0.5	
²²⁸ Radium	15262-20-1	1	0.5	
²⁴⁴ Curium	13981-15-2	1	0.5	
²³⁷ Neptunium	13994-20-2	1	0.5	
²³⁰ Thorium	14269-63-7	1	0.5	
²³² Thorium	7440-29-1	1	0.5	
¹³⁴ Cesium (by gamma)	13967-70-9	1	0.5	
¹³⁷ Cesium (by gamma)	10045-97-3	1	0.5	

TABLE 2
Required Detection Limits for Air Filters

Isotope	MDA (per sample in microCuries)	Approx Sample Volume (Cubic Meters)	Approx. MDA (per unit vol or mass in microCuries/ml)
Effluent Airs	6.2×10^{-8}	7340	$.008 \times 10^{-15}$
Pu-238,239+240	5.7×10^{-7}	7340	$.08 \times 10^{-15}$
Am-241	1.0×10^{-7}	7340	$.01 \times 10^{-15}$
Tritium	2.5×10^{-5}	1.4	$16,000 \times 10^{-15}$
Beryllium	2.5×10^{-1}	7340	3.0×10^{-5} (micrograms/cubic meter)
Total Long-lived Alpha	3.2×10^{-13}	160	0.002×10^{-12}
Ambient Airs			
Pu-238,239+240	1.2×10^{-7}	29,000	$.004 \times 10^{-15}$

It is anticipated that americium and uranium isotope specific analyses will be added to ambient air analyses. Detection limits will be supplied when these analyses are added.

Total long-lived alpha analyses are conducted on individual filters.
Isotopic and beryllium analyses are conducted on composites of filters.
Filters will be composited at the Rocky Flats Plant.

Analysis results are due according to the following schedule:

Total long-lived alpha	Maximum of 1 week from date of collection
Pu, U, Be	Reported 1 month after collection cutoff date
Am	Reported 2 months after collection cutoff date
Tritium	Reported by 3rd Friday of month following collection cutoff date

Collection cutoff dates are the 3rd Thursday and Friday of the month.

TABLE 3

Radiochemistry Reports/Deliverables

<u>Item</u>	<u>No. Copies</u>	<u>Schedule</u>	<u>Distribution</u>
A. Data in Computer Readable Form (Diskette)*	2 (diskettes)	61 days after VTSR of last sample in SDG	EMAD Analysis and Modeling Group
B. Supporting Documentation Package	1 (original)	Completed no later than 66 days after VTSR of last sample in SDG/batch	Retained by laboratory
C. Sample Data Documentation Package	1	66 days after VTSR of last sample in SDG/batch	Validation Subcontractor
D. Written Standard Operating Procedures and QA/QC Plan	1	15 days prior to Pre-Award audit (will be returned at time of audit)	Radioanalytical Program Chemist
E. Controlled copies of Written Standard Operating Procedures	2	30 days after contract award	1. Radioanalytical Program Chemist 2. Validation Subcontractor
F. Controlled copy of QA/QC Plan	1	30 days after contract award	Radioanalytical Program Chemist

VTSR = Validated Time of Sample Receipt

SDG = Sample Delivery Group/Batch

*Data in Computer Readable Form - Electronic data must be supplied for each SDG/batch in a format compatible with EM Department Data Management Systems. Specifications for the type and format of electronic data are included in the specification included in Exhibit IV.

III. RADIOCHEMISTRY REQUIRED DELIVERABLES

Deliverables are those items that must be produced and provided by the subcontractor to EG&G or its designates. These deliverables are described in Table 3. Specifications for the deliverables identified in Table 3 are described below.

A. Sample Data Documentation Package

A radiochemistry sample data package is developed for each SDG.

The radiochemistry sample data package consists of the following items:

1. Cover sheet/transmittal letter.
2. Case narrative.
3. Data summary forms to include:

sample results; blank, duplicate, LCS observed and expected value, replicate, and standard results; MDAs; all deliverable raw data identified in Exhibit I; field and lab sample number cross-references.

4. Copies of items listed on the data checklists found in Exhibit II.

B. Supporting Documentation Package

The supporting documentation package is organized by SDG and is maintained on-site in the laboratory. The supporting documentation package consists of the following:

1. Sample receipt, storage, tracking, document control, and chain-of-custody documents specified in Exhibit III (Chain-of-Custody and Document Control).
2. Originals of the items listed on the data checklists found in Exhibit II (Data Checklists).
3. Standards preparation logbooks (shall demonstrate traceability of stock solutions and working standards to primary standard reference materials).
4. Sample preparation logbooks and benchsheets (shall include method references, sample numbers, analysts' signatures, and dates).
5. Instrument run logs and maintenance logs - one set for each instrument (shall indicate the exact order that standards, calibrations, and samples were counted; sample and lab numbers; analysts' signatures; run times; and dates).
6. MDAs for Radiochemistry.
7. Signature list for all laboratory personnel working on the project (includes typed name, initials, title, and handwritten signature and initials).
8. Document Inventory list for each SDG/Batch.

This documentation shall be maintained in an organized manner allowing for retrieval and reproduction of any of these items. Such retrieval and reproduction will be necessary to perform data review and validation functions and to respond to requests for production of documents for use in legal proceedings.

C. Written Standard Operating Procedures

No later than 15 working days prior to the pre-award audit, the subcontractor will provide to EG&G one copy of the laboratory's SOP's. No later than 30 days after the contract award, the laboratory shall supply controlled copies of the SOPs as follows: one controlled copy to EG&G and one controlled copy to the validation subcontractor. These SOPs shall be specific to the laboratory and be adapted specifically to analysis of EG&G RFP samples. **Generic and/or general operations SOPs are not acceptable.**

SOPs shall cover the following areas in sufficient detail and reflect actual operating conditions in effect during analysis of EG&G RFP samples:

1. Sample receipt and log-in.
2. Sample storage and security.
3. Facility security.
4. Sample tracking (from receipt to sample disposition).
5. Sample analysis methods and references.
6. Data reduction, verification, and reporting.
7. Document control.
8. Data package assembly.
9. Training Records of personnel and resumes.
10. Preparation of standards.
11. Equipment maintenance and calibration.
12. List of instrumentation and equipment (include date purchased, date installed, model number, manufacturer, and service contracts, if any).
13. MDAs.
14. Acceptance criteria for Radiochemistry analyses.
15. Percent Recoveries.
16. Radiochemistry Calculations and Equations.

D. Disposition of Documentation and Samples

Documentation and records generated by the subcontractor shall be retained on-site by the subcontractor for a period of five calendar years. After this period, records may be disposed of with the following provision:

Prior to six months of the date the subcontractor intends to dispose of documentation and records related to EG&G sample analyses, the subcontractor shall notify the EG&G purchasing agent, or designee, in writing. EG&G retains the right to require physical production of the documentation and records by the subcontractor at any time.

Samples and extracts analyzed by the subcontractor that meet EPA's definition of "mixed waste" may be returned to EG&G for disposition. It is anticipated that most EG&G samples will not meet this definition. Disposition of non-mixed waste residual samples and extracts is the responsibility of the laboratory. Prior to disposition of any EG&G samples, EG&G shall be notified in writing.

IV. Technical Audits

For the duration of the contract, EG&G may conduct up to two on-site technical audits per calendar year (at each subcontractor's facility and subtier subcontractor facilities) for the purpose of verifying adherence to quality assurance/quality control requirements and determining the effectiveness of the QA/QC requirements as implemented for analysis of EG&G samples. Pre-award technical audits may be conducted for additional work requested prior to initiation of such work. The pre-award technical audits shall not be counted as one of the two routine technical audits. EG&G shall provide reasonable advance notice in writing to affected laboratories for scheduling technical audits.

GLOSSARY

GLOSSARY

- Absolute Efficiency** - The number of pulses recorded divided by the number of photons emitted by the source.
- Abundance** - The number of photons of a specific energy emitted per 100 atom decays.
- Accuracy** - A measure of the degree of conformity of the mean value with the true value obtained by using a specific method.
- Activity** - The rate of decay of a radioactive source. The rate of decay law is given as:

$$\frac{dN}{dt} = -\lambda N$$
 where λ is defined as the decay constant of the particular radioactive isotope;
 $\lambda = \ln 2 / \text{half-life}$; and N is the number of radioactive nuclei.
- Alpha Particle** - A ${}^4\text{He}$ nucleus emitted by nuclei undergoing alpha decay. Most alpha particle energies of interest to RFP are limited to between 4 and 6 MeV.
- Background** - Radioactive counts detected by the instrument which arise from environmental sources or sources other than the sample being analyzed such as interfering isotopes within the sample, detector contamination, electronic noise, and cosmic rays.
- Background Water** - Tritium-free water or deep well water. Tritium is measured relative to the background water. This measurement is used to determine the net CPM used to calculate the tritium activity. The background activity is known historically to be tritium-free.
- Batch** - A sample delivery group (SDG) or number of samples analyzed together containing the requisite number of reagent blanks, lab control samples, and replicates.
- Becquerel (Bq)** - One Disintegration Per Second. One Bq = 27 pCi.
- Beta Particle** - Fast electron emitted by nuclei undergoing beta decay.
- Branching Ratio** - The number of atom decays per 100 atom decays by a particular mode (i.e. gamma, beta, alpha, etc.)
- Calibration** - (initial) The semi-annual to annual procedure by which an instrument is set up to perform at peak efficiency and sensitivity for radioactive counting.
 (continuing) The semi-weekly to weekly check to see that the peak efficiency and sensitivity levels of the instrument have not changed from the annual initial calibration settings.
- Carrier** - A quantity of non-radioactive or non-labeled material of the same chemical composition as its corresponding radioactive or labeled counterpart.

- Checksource - A radioactive source which is used to confirm the continuing satisfactory operation of an instrument.
- Cocktail - The solution in which samples are placed for measurement in a Liquid Scintillation Counter (LSC). The solution is made up of solvents and scintillators.
- CPM - Counts per minute.
- Curie - 3.7×10^{10} Disintegrations Per Second (DPS)
- Disintegrations - Per Second (DPS) - The number of times a radioactive element undergoes radioactive decay in one second.
- Disintegrations - Per Minute (DPM) - The number of times a radioactive element undergoes radioactive decay in one minute.
- Efficiency - The number of Counts Per Minute (CPM) registered on an instrument divided by the Disintegrations Per Minute (DPM) value of the standard being used to check efficiency; expressed as a percentage.
- Electron Volt (eV) - The kinetic energy gained by a particle carrying an electric charge equal to one electron when it is accelerated through an electric potential difference of 1 volt;
 $1 \text{ eV} = 1.603 \times 10^{-12} \text{ erg.}$
- Energy Resolution - Peak Full Width at Half Maximum (FWHM) (in KeV) divided by the energy of the peak in the centroid channel; expressed as a percentage.
- External Standard - A radioactive source placed adjacent to the liquid sample to produce scintillations in the sample for the purpose of monitoring the level of quenching in the sample.
- Full Width at Half Maximum (FWHM) - The width of the distribution at a level that is just half the maximum ordinate of the peak.
- Half-Life - The time required for one half of the initial number of radioactive nuclei to undergo radioactive decay.
- Intrinsic Efficiency - The number of pulses recorded divided by the number of photons incident on the detector.
- Isotope - One of a number of specific atoms with identical atomic numbers but with different atomic weights, or similarly specific atoms whose nuclei have the same number of protons but different numbers of neutrons.
- (KeV) - kilo electron volt = 10^3 electron volts.
- Laboratory Blanks - Used to determine the existence and magnitude of contamination in the sample preparation process, and to monitor instrument background contributions. A laboratory blank is a full aliquot size of deionized water processed in the same manner as the samples.

Lab Control Sample (LCS) - Any QA, QC, internal standard, measurement control sample, or EPA crosscheck sample which is included in the daily analysis of samples to determine laboratory accuracy. It is aliquotted and analyzed in the same manner as the samples and has a known value. Laboratory accuracy is determined by comparing the known value with the observed value plus or minus the 2σ uncertainty limits of the observed value.

Line Intensity - (See also Line Intensity) - The number of photons of a specific energy emitted per 100 atom decays.

(MeV) - million electron volts = 10^6 electron volts.

Micro Curie (μCi) - 10^{-6} Curies.

Minimum Detectable Activity (MDA) - Health, Safety and Environmental Laboratories have adopted the following definition for detection limit, as given by Harley, 1972:

"The smallest amount of sample activity using a given measurement process (i.e., chemical procedure and detector) that will yield a net count for which there is confidence at a pre-determined level that activity is present."

Multichannel Analyzer (MCA) - A digital computer which collects pulse heights from a detector system as counts; distributes them to proper channel numbers (or addresses) of the pulses; and stores them in a core memory unit.

Nuclide - General term applied to all isotopes of all elements including stable and radioactive forms. (not an isotope)

pico Curie (pCi) - 10^{-12} Curies or 2.22 DPM

Precision - An expression of the repeatability or reproducibility of a measurement.

Preparation Blanks - Water that has been distilled in the tritium distillation apparatus, whose activity is known historically and is used to assure that there is no carry-over or cross-contamination during the distillation step of the sample preparation.

Pulse Height Analysis (PHA) - The process of sorting successive signal pulses from a detector system into parallel amplitude channels of a Multichannel Analyzer (MCA).

QIP (Quench Indicating Parameter) - Value indicating the level of quenching in a sample, may be tSIE, SIS, or H#.

Quenching - The interference with the conversion of decay energy to electronic signal in the photomultiplier tubes usually resulting in a reduction in counting efficiency.

Quench Monitor - The value obtained by the instrument indicating the level or degree of quenching in the sample.

Radionuclide - Any radioactive isotope of an element.

Region of Interest (ROI) - A number of channels selected corresponding to the peak of the radionuclide being measured.

Relative Percent Error - The absolute difference between the observed value and the expected value divided by the expected value.

Self-Absorption - The absorption of radiation, emitted by radioactive atoms, by material in which the radioactive atoms are located.

Standard Deviation (σ) - Measure of dispersion about a mean value of a series of observations expressed in the same units as the mean value.

Standard Statistical Test - (1) Chi-squared test: defined in Knoll, 1979, *Radiation Detection and Measurement*, as:

$$\chi^2 = \sum_{i=1}^N \frac{(X_i - X_e)^2}{X_e}$$

where: $\chi^2 = P$ = the probability from a table in a standard textbook on statistics with acceptable limits from 0.1 to 0.9 and from which a "perfect" fit to the Poisson distribution for large samples would yield a probability of 0.5.

X_i = observed value of i observation.

X_e = average value of all observations

(2) T - test: defined in Mendenhall et. al., *Mathematical Statistics with Applications*, as:

$$T = \frac{X_e - X_i}{s / \sqrt{n}}$$

where: T = values from a "Table of Percentage Points of the t-Distribution" for a given number of observations, average value, and probability for hypothesis testing found in a standard textbook on statistics.

X_i = observed value of observation i

X_e = average value of all observations

s = standard deviation of the observed value

Uncertainty - The error associated with the measurement of the activity of a radioactive isotope which takes into account the random nature of the decay process and the finite count duration.

Weighted Average - Statistical technique of weighting the observed activities for sample and replicate (e.g. weight factor for sample B, $W_B = 1/\sigma_B^2$ where $\sigma_B = 1$ standard deviation and similarly, weight factor for sample B (replicate), $W_B(\text{rep}) = 1/\sigma_B^2(\text{rep})$). The weighted average M_W is calculated as follows: $M_W = \sum w_i M_i / \sum w_i$, where M_i = activity of sample or replicate and w_i = weighting factor of sample or replicate.

EXHIBIT I

RADIOCHEMISTRY ANALYTICAL REQUIREMENTS

- Requirements for Isotopic Analyses By Alpha Spectrometry
- Requirements for Tritium Analyses By Liquid Scintillation
- Requirements for Isotopic Analyses By High Resolution Gamma Spectrometry
- Requirements for Gross α and β Analyses By Gas Proportional Counting
- Requirements for $^{226}\text{Radium}$ Analyses by Radon Emanation
- Requirements for Radiometric Strontium, Cesium and $^{228}\text{Radium}$ Analyses by Gas Proportional Counting

REQUIREMENTS FOR ISOTOPIC ANALYSES BY ALPHA SPECTROMETRY

EG&G Rocky Flats
Environmental Management Department

The following are requirements for isotopic analyses using Alpha Spectrometry. These requirements address instrumentation, equipment used to prepare samples, sample deposition methods, calibrations, sample holding times, case narratives, quality control including LCSs, replicate samples, laboratory blanks, MDAs, chemical recovery, instrument efficiency, aliquot sizes, documentation for calculations, and raw data.

I. General Requirements

The General Requirements section describes the specifications for instrumentation, calibrations, sample holding times, and Case Narratives.

1. Instrumentation

- 1.1 The alpha detector system shall consist of any detector suitable for measuring the alpha isotopes of interest in the range from 4 to 6 MeVs. The maximum range covered shall be from 3 to 7 MeVs for 512 channels of memory.
- 1.2 The detector system shall have a resolution of no higher than approximately 100 KeV for an ^{241}Am source of 5.49 MeV alpha, or a ^{239}Pu source of 5.14 MeV alpha. The resolution is defined as the product of system gain (in KeV/channel) times the channels Full Width at Half Maximum (FWHM in channels).
- 1.3 The percent resolution (FWHM value of the tracer peak in KeVs divided by the tracer isotope energy in KeVs) shall be equal to or less than 2%.
- 1.4 The laboratory shall identify and document the instrument manufacturer, model, instrument configuration, program and instrument settings, detectors IDs and any changes or modifications in the instrument maintenance logs.
- 1.5 The detector system should be set up according to the manufacturer's instructions so that the MDAs meet the RDLs for the samples.
- 1.6 The instrumentation shall be surge protected.
- 1.7 The method used to deposit a sample for counting by alpha spectrometry shall be equivalent to, or superior to, electroplating.
- 1.8 Near-background to low-level samples (≤ 100 pCi total sample activity) shall be segregated from intermediate level samples (> 100 pCi but ≤ 10 nCi total sample activity) which shall be segregated from high level samples (> 10 nCi total sample activity); and each of these groups shall have its own electroplating apparatus.

- 1.9 The metal disc cathodes used in the electroplating apparatus shall be flat and uncupped discs, made of stainless steel (or other metal with equivalent or superior electroplating properties), and be scrupulously cleaned and degreased prior to use.
- 1.10 The resin in the ion exchange columns shall be replaced with fresh resin prior to each use.

2. Calibrations

- 2.1 The standards used for energy calibrations and efficiency checks in alpha spectrometry shall be isotopes of Am or Pu or Cm or U of known energy. (Use of isotopes other than these four may be considered; however, concurrence of EG&G shall be obtained prior to use.) The standard material shall be NIST-traceable or equivalent standard reference materials, and be of the highest purity obtainable. Certificates shall be submitted on a one time basis or as requested.
- 2.2 The standard material used in tracer solutions for isotopic dilutions shall be NIST-traceable, or equivalent, and be of the highest purity obtainable. Tracer spike aliquots shall have DPM values consistent with the activity of the samples. Certificates and traceability documentation including dilution logs shall be submitted on a one time basis or as requested.
- 2.3 The standards for energy calibration and efficiency checks shall be counted at least once a week in each detector. The system gain (KeV/channel) is based on the known energy of the standard nuclides used for calibration and the centroid channel number of their observed peaks. The efficiency of the system is determined by counting the standard of known activity and dividing CPMs obtained by the known DPMs of the standard. System efficiency and system gain data shall be submitted on a quarterly basis and submitted when instrument settings are changed.
- 2.4 The activity, preparation and any changes to or replacement of the tracer solutions shall be submitted with each SDG. Tracer solution preparations shall have an overall uncertainty equal to or less than 2% of the uncertainty value for the reference material.
- 2.5 An instrument background shall be measured at least once a month or more frequently showing background counts obtained in each Region of Interest (ROI) for at least a 12 hour count duration and shall be submitted on a monthly basis.
- 2.6 Samples shall be counted for durations long enough to achieve the RDLs or for a maximum of 1000 minutes.
- 2.7 Standards used for determining efficiency shall be counted for a minimum of 2000 counts.

3. Sample Holding Times

- 3.1 Water samples shall be analyzed within 180 days from date of collection.

4. Case Narratives

- 4.1 Case narratives shall be specific for each SDG as to abnormalities encountered with samples. Reasons shall be given why proper aliquot size was not used, or if RDLs were not met. Matrix problems, poor counting precision, or poor accuracy shall be explained. Reanalyses shall be explained and the analytical results that are reported shall be identified with their respective reanalysis data. Deviations from SOPs shall be explained.

II. Quality Control Requirements

The Quality Control Requirements section describes the specifications for LCSs, replicate analyses, laboratory blank analyses, determining MDAs, chemical recovery criteria, and aliquot sizes.

1. Laboratory Control Samples

- 1.1 LCSs shall be analyzed at a frequency of 5% per batch.
- 1.2 The LCS activity shall be from greater than 5x to less than or equal to 50x the RDL.
- 1.3 LCSs shall be prepared and analyzed in the same manner as the samples.
- 1.4 LCSs shall have the same aliquot size as the samples.
- 1.5 LCSs shall have the same RDLs as the samples.
- 1.6 Using the Alpha Spectrometry Overall Counting Uncertainty (Section III 3.2) the observed value of the LCSs shall be within 3σ control limits of the expected LCS value and shall have a relative percent error that does not exceed 10%.
- 1.7 LCSs shall be counted for the same count durations as the samples.
- 1.8 LCS data shall be submitted with each data package and shall include the expected values for all isotopes for which the samples are being analyzed.
- 1.9 An LCS with a deionized water matrix may be used as a LCS for samples with matrices other than that of water.

2. Replicate Analyses

- 2.1 Replicate analyses shall be analyzed at a frequency of 10% per batch.
- 2.2 Replicate samples shall be prepared and analyzed in the same manner as the samples.

- 2.3 Replicate samples shall have the same aliquot size as the samples.
- 2.4 Replicate samples shall have the same RDLs as the samples.
- 2.5 Replicates shall be counted for the same count durations as the samples.
- 2.6 Replicate analyses data shall be submitted with each data package.
- 2.7 The replicate analyses shall be within the 3σ range of the weighted average and its associated standard error. "Hot" particles may be present in soils, sediments, and total waters and this will be taken into consideration when evaluating duplicates. See the Glossary for the Weighted Average formula.

3. Laboratory blanks

- 3.1 Laboratory blanks shall be analyzed at a frequency of 5% per batch.
- 3.2 Laboratory blanks shall be prepared and analyzed in the same manner as the samples.
- 3.3 Laboratory blanks shall have the same aliquot size as the samples.
- 3.4 Laboratory blanks shall be counted for the same count duration as the samples.
- 3.5 Deionized water may be used as a laboratory blank for soil samples.

4. Minimum Detectable Activities

- 4.1 Count durations for samples, replicates, blanks, and backgrounds shall be optimized so that the MDAs achieve the RDLs. Interferences, contaminants, and other matrix problems may cause the sample MDAs to exceed the desired MDAs; however, the laboratory shall demonstrate (usually by reanalysis) that the MDA could not be met due to the matrix and not because of inadequate count time, laboratory problems, or other limitations. Reanalysis due to matrix problems will be treated as an additional sample analysis. In all cases, MDAs which fail to achieve the required RDL shall be fully explained in the Case Narratives.
- 4.2 The MDAs shall be reported on the sample calculations sheet. The last background count taken (1 month old or less) shall be used for calculations.
- 4.3 The laboratories shall compile quarterly a history of RFP laboratory blanks used to perform the sample analyses. The analytical results shall be submitted on a quarterly basis.

5. Chemical Recovery

- 5.1 Chemical recovery for U analyses shall be $>30\%$ but $<105\%$. Chemical recovery for Pu and Am analyses shall be $>20\%$ but $<105\%$. Chemical recoveries outside these limits require the affected samples to be reanalyzed.

- 5.2 Chemical recovery shall be calculated based on the latest instrument efficiency value.
- 5.3 Counts obtained for the tracer peak, DPMs of tracer used, and aliquot of tracer used shall appear in the raw data.

6. Aliquot Size

- 6.1 The aliquot size shall be optimized to achieve the RDLs. If the RDLs are not achieved and the aliquot sizes are less than 0.5 Liter for U, and 1.0 Liter for Am, or 1.0 Liter for Pu, then the problem shall be addressed in the Case Narrative.

III. Raw Data and Documentation

The Raw Data and Documentation section describes the specifications for calculations sheets, spectra, calculating MDAs, and submittal of standard operating procedures.

1. Calculation Sheets

- 1.1 Alpha Spectrometry calculation sheets shall include: batch numbers, sample ID numbers, detector ID numbers, LCS ID numbers and results, expected values of the LCSs, laboratory blank results, aliquot of samples, tracer name and DPM values, sample and background count durations, chemical recoveries, instrument efficiencies, ROIs and FWHM values for tracers, the ROIs for the isotopes of interest, analysis date, sample activity, uncertainty values, and MDAs in appropriate units (see Scope of Work Section B.2.16), and any relevant comments on quality of the analysis.
- 1.2 A summary report data section shall include: batch numbers, sample IDs, sample collection dates, activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.14).

2. Hard Copies of Spectra

- 2.1 Either an x-y plot or channel-by-channel counts printout of the alpha spectra shall be included in each SDG for every sample, LCS, laboratory blank, and replicate.
- 2.2 The laboratory shall submit raw data, instrument program printouts which include: sample ID numbers, count dates, sample and background count durations, and instrument ID numbers.

3. Calculations

- 3.1 The MDA shall be calculated as follows:

$$\begin{array}{lcl} \text{MDA (pCi/Aliquot in} & = & \frac{4.65 S_B + 2.71/TEY}{a * \text{Aliquot}} \\ \text{appropriate units} & & \\ \text{See Scope of Work} & & \\ \text{Section B.2.16)} & & \end{array}$$

Where S_B = Standard deviation of the population of quarterly RFP blank values (DPM)
 T = Sample count duration in minutes
 E = Detector efficiency
 Aliquot = Aliquot in appropriate units (see Scope of Work, Sec.B.2.16)
 Y = Chemical Recovery for the sample
 a = 2.22 conversion for DPMs to pico Curies

3.2 Alpha Spectrometry Overall Counting Uncertainty shall be calculated using:

Preliminary Calculations:

$$R_s = [R_{s+b} - R_b] = \left[\frac{C_{s+b}}{T_s} - \frac{C_b}{T_b} \right] \quad S_{R_s} = \sqrt{\frac{C_{s+b}}{T_s^2} + \frac{C_b}{T_b^2}}$$

$$R_{ts} = [R_{ts+tb} - R_{tb}] = \left[\frac{C_{ts+tb}}{T_{ts}} - \frac{C_{tb}}{T_{tb}} \right] \quad S_{R_{ts}} = \sqrt{\frac{C_{ts+tb}}{T_{ts}^2} + \frac{C_{tb}}{T_{tb}^2}}$$

$$\frac{Dpm}{Alq} = \frac{R_s * T_{dpm}}{R_{ts}}$$

Uncertainty calculation $\left(S \frac{Dpm}{Alq} \right)$ is:

$$S \frac{Dpm}{Alq} = \left(\frac{Dpm}{Alq} \right) \sqrt{\left(\frac{S_{R_s}}{R_s} \right)^2 + \left(\frac{S_{R_{ts}}}{R_{ts}} \right)^2}$$

Where:

C_{s+b} = Observed number of counts for the sample.
 T_s = Count times for the sample.
 C_b = Observed number of counts for the sample background ROI.
 T_b = Count times for the sample background.
 C_{ts+tb} = Observed number of counts for the tracer ROI.
 T_{ts} = Count times for the tracer.
 C_{tb} = Observed number of counts for the tracer background ROI.
 T_{tb} = Count times for the tracer background.
 R_s = Count rate of the sample.
 S_{R_s} = Standard deviations of the sample count rate.
 R_{ts} = Count rate of the tracer.
 $S_{R_{ts}}$ = Standard deviations of the tracer count rate.
 T_{dpm} = Activity of tracer added to the sample.

4. Equipment

4.1 The laboratory shall calibrate non-class A pipets on a quarterly basis to ensure that standard aliquots are delivered. The calibration data shall be available on an as requested basis and be retained by the laboratory for on-site technical audits.

Where S_B = Standard deviation of the population of quarterly RFP blank values (DPM)
 T = Sample count duration in minutes
 E = Detector efficiency
 Aliquot = Aliquot in appropriate units (see Scope of Work, Sec.B.2.16)
 Chem Rec = Chemical Recovery for the sample
 a = 2.22 conversion for DPMs to pico Curies

3.2 Alpha Spectrometry Overall Counting Uncertainty shall be calculated using:

Preliminary Calculations:

$$R_s = [R_{s+b} - R_b] = \left[\frac{C_{s+b}}{T_s} - \frac{C_b}{T_b} \right] \quad S_{R_s} = \sqrt{\frac{C_{s+b}}{T_s^2} + \frac{C_b}{T_b^2}}$$

$$R_{ts} = [R_{ts+tb} - R_{tb}] = \left[\frac{C_{ts+tb}}{T_{ts}} - \frac{C_{tb}}{T_{tb}} \right] \quad S_{R_{ts}} = \sqrt{\frac{C_{ts+tb}}{T_{ts}^2} + \frac{C_{tb}}{T_{tb}^2}}$$

$$\frac{Dpm}{Alq} = \frac{R_s * T_{dpm}}{R_{ts}}$$

Uncertainty calculation $\left(S \frac{Dpm}{Alq} \right)$ is:

$$S \frac{Dpm}{Alq} = \left(\frac{Dpm}{Alq} \right) \sqrt{\frac{S_{R_s}^2}{R_s^2} + \frac{S_{R_{ts}}^2}{R_{ts}^2}}$$

Where:

C_{s+b} = Observed number of counts for the sample.
 T_s = Count times for the sample.
 C_b = Observed number of counts for the sample background ROI.
 T_b = Count times for the sample background.
 C_{ts+tb} = Observed number of counts for the tracer ROI.
 T_{ts} = Count times for the tracer.
 C_{tb} = Observed number of counts for the tracer background ROI.
 T_{tb} = Count times for the tracer background.
 R_s = Count rate of the sample.
 S_{R_s} = Standard deviations of the sample count rate.
 R_{ts} = Count rate of the tracer.
 $S_{R_{ts}}$ = Standard deviations of the tracer count rate.
 T_{dpm} = Activity of tracer added to the sample.

4. Equipment

- 4.1 The laboratory shall calibrate non-class A pipets on a quarterly basis to ensure that standard aliquots are delivered. The calibration data shall be available on an as requested basis and be retained by the laboratory for on-site technical audits.

REQUIREMENTS FOR TRITIUM ANALYSES BY LIQUID SCINTILLATION

EG&G Rocky Flats
Environmental Management Department

The following are requirements for tritium analyses using Liquid Scintillation (LS). These requirements address instrumentation, calibrations, sample holding times, case narratives, quality control including LCSs, replicate samples, background water, preparation blanks, MDAs, quench monitor values, documentation for calculations, and raw data from counters.

I. General Requirements

The General Requirements section describes the specifications for instrumentation, initial and continuing calibrations, sample holding times, Case Narratives, and sample preparation.

I. Instrumentation

- 1.1 A low-background counter consisting of two photomultiplier tubes which recognize coincident events is required. The signal is digitized and stored as a count in a multi-channel analyzer.
- 1.2 The performance of the instrument in the high sensitivity count mode, figure of merit (FOM), shall be maximized when the samples are counted.

$$\text{FOM} = E^2/B \quad \begin{array}{l} \text{Where } E = \text{detector efficiency in percent} \\ \text{Where } B = \text{background CPM} \end{array}$$

The window setting shall be set at the point where E^2/B is the highest number. The optimized window setting is used for sample counting.

- 1.3 Counter efficiency, aliquot size, and count duration shall be optimized so that the MDAs meet the RDLs for the samples.
- 1.4 The laboratory shall identify the instrument manufacturer, model, instrument configuration, program and instrument settings, quench monitor used, and date of installation.
- 1.5 The instrumentation shall be surge protected.

- 1.6 The laboratory should set up instrumentation according to instrument manufacturers' instructions, and document any changes or modifications.

2. Calibrations

- 2.1 The laboratory should follow the instrument manufacturers' instructions for initial and continuing calibrations using appropriate unquenched sealed standards as check sources and to calibrate instrument detectors.
- 2.2 The laboratory shall calibrate instrumentation weekly, or before running each batch, with appropriate unquenched flame-sealed NIST-traceable standards. The laboratory shall maintain a log of calibration data on each detector including calibration date, activity with uncertainty values of unquenched flame-sealed standards, certification and expiration dates on the standards, and CPMs obtained on each date. A copy of this data or the control chart shall be submitted with each SDG. The standard shall be counted to a low counting error (at least 100,000 accumulated counts). The quench monitor value obtained when the standard is counted shall be available and shall be consistent with instrument manufacturer specifications.
- 2.3 The level of quenching shall be verified using quench curves, internal standardization spikes, quench monitor values, and/or using external standard methods. The laboratory shall submit the pictorial printout of the quench curve including an efficiency/QIP table and the data used to generate the curve with each SDG.
- 2.4 The aliquot size added to the counting cocktail shall be the same for the efficiency standards and the samples. Efficiency measurements shall be taken weekly or before running each batch.
- 2.5 The laboratory shall maintain long-term efficiency measurements taken over three months contiguous to the sample count date identifying the 3σ uncertainty limits based on long-term efficiency measurements and shall submit this data on a quarterly basis. Alternatively, the laboratory shall maintain a log of detector efficiency which includes uncertainties and quench monitor values and submit this data on a quarterly basis.
- 2.6 Instrument backgrounds shall be taken weekly, or before running each batch. The laboratory shall maintain, on a quarterly basis, long-term instrument background measurements taken over three months contiguous to the sample count date identifying the 3σ uncertainty limits based on long-term background measurements. This data shall be submitted with each data package and include the quench monitor values.
- 2.7 The laboratory shall perform standard statistical tests as referenced in the Glossary to determine instrument reliability and submit the results on a quarterly basis.

- 2.8 Calibration data shall be retained for on-site technical audits. The activity with uncertainty values, certification dates, calibration dates, CPMs, and expiration dates of the standards used to calculate the detector efficiency shall be included.

3. Sample Holding Times

- 3.1 Water samples shall be analyzed within 180 days from date of collection.

4. Case Narrative

- 4.1 Case narratives shall be specific for each SDG as to abnormalities encountered with samples. Reasons shall be given why proper aliquot size was not used, or if RDLs were not met. Matrix problems, poor counting precision or poor accuracy shall be explained. Reanalyses shall be explained and the analytical results that are reported shall be identified with their respective reanalysis data. Deviations from SOPs shall be explained.

5. Sample Preparation

- 5.1 All LCSs, blanks, preparation duplicates, replicates, and samples shall be distilled prior to analysis. A copy of the distillation log shall be submitted with each SDG.
- 5.2 Samples shall be counted in vials equivalent, or superior to, low potassium glass vials or high density polyethylene vials. When polyethylene vials are used the samples should be counted within three days (but absolutely shall be counted in less than 7 days) after the cocktail was placed in the vial.
- 5.3 Samples shall be mixed with the cocktail and "dark adapted" at least thirty minutes, or according to cocktail manufacturer's instructions before counting.

II. Quality Control Requirements

The Quality Control Requirements section describes the specifications for analyzing LCSs, replicate samples, and background water and preparation blanks, and determining MDAs, and quench monitor values.

1. Laboratory Control Samples

- 1.1 LCSs shall be analyzed at a frequency of 5% per batch.
- 1.2 The LCS activity shall be from greater than 5x or equal to 30x the instrument RDL.
- 1.3 LCSs shall be prepared, distilled and analyzed in the same manner as the samples.
- 1.4 LCSs shall have the same aliquot size added to the counting cocktail as the samples.

- 1.5 Using the statistical counting error, the observed LCS value shall be within 3σ control limits of the expected LCS value and shall have a relative percent error, not to exceed 15%.
- 1.6 LCSs shall be counted for the same count durations as the samples.
- 1.7 LCS data shall be submitted with each data package with the expected values. Quench monitor values shall be included.

2. Replicate Analyses

- 2.1 Replicate analyses shall be analyzed at a frequency of 10% per batch.
- 2.2 Replicate samples shall be prepared, distilled and analyzed in the same manner as the samples.
- 2.3 Replicate samples shall have the same aliquot size added to the counting cocktail as the samples.
- 2.4 Replicate samples shall have the same RDLs as the samples.
- 2.5 Replicates shall be counted for the same count durations as the samples.
- 2.6 Replicate analyses data shall be submitted with each data package and include the quench monitor values.
- 2.7 The replicate analyses shall be within 3σ the range of the weighted average and its associated standard error. See the Glossary for the Weighted Average formula.

2. Background water and Preparation blanks

- 3.1 The laboratory shall identify the source water used for background water. Laboratories should make an effort to obtain certified "EPA dead water" for preparing background water vials.
- 3.2 Preparation blanks shall be analyzed at a frequency of 5% per batch.
- 3.3 Preparation blanks shall be prepared, distilled and analyzed in the same manner as the samples.
- 3.4 Preparation blanks shall have the same aliquot size added to the counting cocktail as the samples.
- 3.5 Background water and preparation blank data shall be submitted with each data package and shall include the quench monitor values. Background water and preparation blanks shall be counted for at least the same count duration as the samples.

4. Minimum Detectable Activities

- 4.1 Count durations for samples, replicates, blanks, and backgrounds shall be optimized so that the MDAs achieve the RDLs. Interferences, high dissolved solids, and other matrix problems may cause the sample MDAs to exceed the desired MDAs; however, the laboratory shall demonstrate (usually by reanalysis) that the MDA could not be met due to the matrix and not because of inadequate count time, laboratory problems, or other limitations. Reanalysis due to matrix problems will be treated as an additional sample analysis. In all cases, failure to achieve the required MDAs shall be fully explained in the Case Narratives.
- 4.2 The MDAs shall be reported on the sample calculations sheet. The last background count taken (1 week old or less) shall be used for calculations.

5. Quench Monitor Values

- 5.1 A reliable method for efficiency determination is the internal standard method. In this procedure, a sample is counted, after which a known amount of NIST-traceable tritiated water (or equivalent standard reference material) is added and the sample is recounted. The additional counts due to the added standard or spike are used to compute the counting efficiency for the sample. The spiked sample shall be counted long enough to acquire 10,000-50,000 counts. The chemical form of the standard must not induce quench and the volume of standard added should be small so as to not alter the sample volume of the initial count significantly. The integrity of the tritiated water solution used for spikes shall be verified with each batch of samples by running laboratory control samples. The stock tritiated water solution prepared from the flame-sealed NIST standard reference material (or equivalent) shall be replaced with a fresh standard every 6 months. The laboratory shall submit a copy of the calibration reference standard certificate from NIST with dilution logbook pages and submit data of the sample and spiked sample including the quench monitor values with each SDG.
- 5.2 The external standard method for efficiency determination uses the quench monitor value as a measure of the degree of quenching in a sample. When the measurement of one standard is used, the efficiency determination should be valid when the quench monitor values of the samples, instrument backgrounds, reagent blanks, and LCSs fall within $\pm 5\%$ of the quench monitor value of the standard used to determine the efficiency. The efficiency standard shall be counted twice. The average of the efficiency shall be used to determine the activity of the samples. The standard does not have to be prepared daily, but shall be replaced if a decrease in efficiency is noted or phase separation is apparent in the cocktail. The integrity of the tritiated water standard used for the efficiency determination shall be verified and the calibration reference standard certificate from NIST shall be submitted on a one time basis or as requested.

- 5.3 The external standard method for efficiency determination can be performed by counting a set of simulated low-level NIST-traceable quenched standards (or equivalent). The results of the curve (efficiency versus quench monitor value) are stored in the instrument. Commercial quench sets accurate within 2-3% can be used or the lab can prepare their own quench set provided that the accuracy is within 2-3%. Repeat measurements of the CPM and quench monitor value will increase the accuracy of the quench curve. The standard quench set shall be counted to a low counting error (less than 1% at 3σ or 100,000 counts) at least twice. The accuracy of the quench curve shall be checked by running known samples using the curve. The efficiency coefficients shall be submitted monthly or as requested.
- 5.4 The laboratory shall demonstrate the quench curve performance either by showing that a NIST-traceable flame-sealed unquenched standard which has been counted weekly has been charted or by showing that a new quench curve was generated monthly using prepared standard vials or certified, unexpired, purchased standard vials. The integrity of the tritiated water standard used for the quench curve shall be verified and the calibration reference standard certificate from NIST shall be submitted on a one time basis or as requested. The laboratory shall submit the pictorial printout of the quench curve including an efficiency/QIP table with the data used to generate the curve.

III. Raw Data and Documentation

The Raw Data and Documentation section describes the specifications for calculations sheets, reporting counting data, calculating MDAs, and standard operating procedures.

1. Calculations Sheets

- 1.1 Tritium calculation sheets shall include: batch numbers; detector background with uncertainty and quench monitor values; sample count dates; detector efficiency with quench monitor values; and aliquot size.
- 1.2 Summary Report data section shall include: batch numbers, sample IDs, sample collection dates and activities, uncertainties, and MDAs in appropriate units (see Scope of Work, Section 3.2.14).
- 1.4 The laboratory shall identify the cocktail used and the scintillation vial used.

2. Data From the Counter

- 2.1 The data submitted from the counter shall be legible and include: detector ID numbers, count dates, Sample IDs, quench monitor values, LCS IDs, background water and preparation blanks, instrument backgrounds, efficiency standards identified, CPMs, sample and background count durations, and printed instrument program settings.

3. Calculating MDAs

3.1 The MDA shall be calculated as follows:

$$\text{MDA (pCi/Aliquot in appropriate units see Scope of Work Section B.2.16)} = \frac{4.66 (BKG/T_1)^{0.5}}{\text{Eff} * \text{Aliquot} * 2.22 * e^{-\lambda t}}$$

Where BKG = Background count rate in CPM
 T_1 = Sample count duration
 Eff = Detector efficiency
 Aliquot = Aliquot in appropriate units (see Scope of Work Sec. B.2.16)
 $e^{-\lambda t}$ = Decay correction (for specific radionuclides)

4. Equipment

4.1 The laboratory shall calibrate non-class A pipets on a quarterly basis to ensure that standard aliquots are delivered. The calibration data shall be available on an as requested basis and be retained by the laboratory for on-site technical audits.

REQUIREMENTS FOR ISOTOPIC ANALYSES BY HIGH RESOLUTION GAMMA SPECTROMETRY

EG&G Rocky Flats
Environmental Management Department

The following are requirements for isotopic analyses using High Resolution Gamma Spectrometry. These requirements address instrumentation, calibrations, sample holding times, case narratives, quality control including LCSs, matrix blanks, MDAs, efficiency versus energy curves, documentation for calculations, and raw data.

I. General Requirements

The General Requirements section describes the specifications for instrumentation, calibrations, sample holding times, and Case Narratives.

1. Instrumentation

- 1.1 The High Resolution gamma detector system shall consist of any germanium-lithium or intrinsic germanium detector suitable for measuring the gamma isotopes of interest in the range from 25 to 2000 KeVs.
- 1.2 The detector system shall have a resolution of 3.0 KeV or less for the Co-60 peak at 1332 KeV. The resolution is defined as the product of system gain (in KeV/channel) times the channels FWHM (in channels).
- 1.3 The laboratory shall identify and document the instrument manufacturer, model, instrument configuration, program and instrument settings, detectors IDs, and any changes or modifications in the instrument maintenance logs.
- 1.4 The detector system should be set up according to the manufacturer's instructions so that the MDAs meet the RDLs for the samples.
- 1.5 The laboratory shall maintain an instrument run log and a maintenance log for each instrument used.
- 1.6 The instrumentation shall be surge protected.

2. Calibrations

- 2.1 The standard materials used to prepare the efficiency curves and checksources, energy calibration curves and checksources, and mixed-gamma sources shall be NIST-traceable or equivalent, unexpired, and be of the highest purity obtainable.

- 2.2 The checksource standards shall be counted at least once per month in each detector and the results shall be submitted on a monthly basis.
- 2.3 The system gain (KeV/channel) is defined as the slope of the detector's energy calibration curve and shall be 1.00 KeV/channel or less. The coefficients for the energy calibration curve shall be shown on the printout and submitted with each SDG.
- 2.4 The characteristic curve of the detector shall be determined by analyzing standards for each sample matrix, mass, and geometry which have strong gamma lines covering the energy range desired. For each energy the observed efficiency is divided by the line intensity and multiplied by 100; thus creating a full intensity efficiencies versus energies curve. The coefficients for the curve shall be shown on the sample printout and submitted with each SDG.
- 2.5 An instrument background spectra shall be measured showing background counts obtained in each peak of interest for a count duration which is at least as long as the sample count duration and the spectral summary shall be submitted on an as requested basis.
- 2.6 Samples shall be counted for durations long enough to achieve the RDLs.
- 2.7 The coefficients for the FWHM shall be shown on the sample printout and submitted with each SDG.
- 2.8 The Co-60 peak for the checksource standard shall not be shifted more than 2 channels from the centroid position.

3. Sample Holding Times

- 3.1 Water samples shall be analyzed within 180 days from date of collection.

4. Case Narratives

- 4.1 Case narratives shall be specific for each SDG as to abnormalities encountered with samples. Reasons shall be given why proper aliquot size was not used, or if RDLs were not met. Matrix problems, poor counting precision or poor accuracy shall be explained. Reanalyses shall be explained and the analytical results that are reported shall be identified with their respective reanalysis data. Deviations from SOPs shall be explained.

II. Quality Control Requirements

The Quality Control Requirements section describes the specifications for LCSs, replicate analyses, matrix blank analyses, determining MDAs, and aliquot sizes.

1. Laboratory Control Samples

- 1.1 LCSs shall be analyzed at a frequency of 5% per batch.
- 1.2 The LCS activity shall not exceed 1000 pico Curies for the total sample activity.
- 1.3 LCSs shall be prepared in the same matrix, geometry, and mass as the samples.
- 1.4 LCSs shall have the same aliquot size as the samples.
- 1.5 LCSs shall have the same RDLs as the samples.
- 1.6 Using the statistical counting error, the observed LCS value shall be within 3σ control limits of the expected LCS value and shall have a relative percent error, not to exceed 5%.
- 1.7 LCS sample data shall be submitted with each data package and shall include the expected values for all isotopes for which the samples are being analyzed.
- 1.8 The activities based on the two confirming lines of the Co-60 in the LCS shall be within the range of the weighted average and its associated error at the 99% confidence limit.
- 1.9 The K-40 peak in the sample spectra shall not drift by more than 3 channels from its centroid channel number.

2. Replicate Analyses

- 2.1 Replicate analyses shall be analyzed at a frequency of 10% per batch.
- 2.2 Replicate samples shall be prepared and analyzed in the same manner as the samples.
- 2.3 Replicate samples shall have the same aliquot size as the samples.
- 2.4 Replicate samples shall have the same RDLs as the samples.
- 2.5 Replicates shall be counted for the same count durations as the samples.

3. Matrix Blanks

- 3.1 Matrix blanks shall be analyzed at a frequency of 5% per batch.

- 3.2 Matrix blanks shall consist of a full aliquot of deionized water that is counted in the same geometry as the samples.
- 3.3 Matrix blanks shall be counted for the same count duration as the samples.
- 3.4 A water matrix blank may be used as a matrix blank for samples with matrices other than that of water.

4. Minimum Detectable Activities

- 4.1 Count durations for samples, replicates, blanks, and backgrounds shall be optimized so that the MDAs achieve the RDLs. Interferences, contaminants, and other matrix problems may cause the sample MDAs to exceed the desired MDAs; however, the laboratory shall demonstrate (usually by reanalysis) that the MDA could not be met due to the matrix and not because of inadequate count time, laboratory problems, or other limitations. Reanalysis due to matrix problems will be treated as an additional sample analysis. In all cases, failure to achieve the required MDAs shall be fully explained in the Case Narratives.
- 4.2 The MDAs shall be reported on the sample calculations sheet. The last background count taken (1 month old or less) shall be used for calculations.

III. Raw Data and Documentation

The Raw Data and Documentation section describes the specifications for calculations sheets, spectra, calculating MDAs, and submittal of standard operating procedures.

1. Calculation Sheets

- 1.1 Gamma Spectrometry calculation sheets shall include: batch numbers, sample ID numbers, LCS ID numbers and results, expected values of the LCSs, matrix blank results, aliquot of samples, sample and background count durations, coefficients of energy calibration curve, coefficients of characteristic efficiency curve, coefficients for FWHM, energies (keV) of peaks of interest, address channels, sample and background net areas (counts), critical level counts, ROIs and FWHM values, the ROIs for the isotopes of interest, analysis date, sample activity, uncertainty, and MDAs in appropriate units (see Scope of Work Section B.2.14), and any relevant comments on quality of the analysis.
- 1.2 A summary report data section shall include: batch numbers, sample IDs, sample collection dates, activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.16).

2. Hard Copies of Spectra

- 2.1 Either a system program printout of the data or channel-by-channel counts printout of the ROIs shall be included for every sample, LCS, matrix blank, and replicate with each SDG.
- 2.2 The laboratory shall submit raw data and instrument program printouts which include: sample ID numbers, count dates, sample and background count durations, and instrument ID numbers.

3. CalculatingMDAs

- 3.1 The MDA shall be calculated as follows:

$$\begin{array}{l} \text{MDA (pCi/Aliquot in appropriate units} \\ \text{see Scope of Work Sec. B.2.16)} \end{array} = \frac{4.66 (BKG/T_1)^{0.5}}{\text{Eff} * \text{Aliquot} * \text{abn} * .037}$$

Where BKG = Background count rate in CPM
 T_1 = Sample count duration
 Eff = Efficiency value at peak energy (from characteristic efficiency curve)
 Aliquot = Aliquot in appropriate units (see Scope of Work Sec. B.2.16)
 abn = Abundance or line intensity (expressed as a decimal)
 .037 = Conversion factor from disintegrations per second (DPS) to pCi

4. Equipment

- 4.1 The laboratory shall calibrate on a quarterly basis non-class A pipets and sample dispensers to ensure that standard aliquots are delivered. The data shall be available on an as requested basis and be retained by the laboratory for on-site audits.

REQUIREMENTS FOR GROSS α AND β ANALYSES BY GAS PROPORTIONAL COUNTING

EG&G Rocky Flats
Environmental Management Department

The following are requirements for Gross α and β analyses using Gas Proportional Counters (GPCs). These requirements address instrumentation, calibrations, sample holding times, case narratives, quality control including LCSs, replicate analyses, laboratory blanks, MDAs, self-absorption factors, aliquot sizes, documentation for calculations, and raw data from counters.

I. General Requirements

The General Requirements section describes the specifications for instrumentation, initial and continuing calibrations, sample holding times, and case narratives.

1. Instrumentation

- 1.1 Instrumentation shall consist of any low-background, anti-coincidence proportional counter consisting of a sample detector, cosmic detector, and pulse height discriminating circuitry for measuring α and β activity, or demonstrated equivalent.
- 1.2 The laboratory shall identify the instrument manufacturer, model, instrument configuration, program and instrument settings, crosstalk values, voltages, dates of installation, and detector identifications.
- 1.3 The laboratory shall set up instrumentation according to the manufacturer's instructions; any changes or modifications thereto shall be documented.
- 1.4 Counter efficiency, aliquot size, and count duration shall be optimized so that the MDAs meet the RDLs for the samples.
- 1.5 The instrumentation shall be surge protected.

2. Initial Calibration

- 2.1 The laboratory shall follow instrument manufacturer's instructions using appropriate sources to set voltages and calibrate instrument detectors.

- 2.2 The laboratory shall submit calibration verification data every three years or whenever re-calibrations are performed. Calibration verification data shall include: radionuclide name; certification, expiration dates, and DPM activities of standards; volumes of standards used; count durations; calibration dates; mg weights of salts; alpha counts obtained; beta counts obtained; efficiencies; alpha/beta crosstalk values, if applicable; and best-fit curve coefficients.
- 2.3 The laboratory shall perform calibrations for each radionuclide to be counted, and the standard reference material shall have the same physical form (size, shape, planchet material, etc.) as the samples to be counted.
- 2.4 For multiple counting systems, calibration equivalency shall be shown for each detector in the array. The complete self-absorption curve maximum allowable weight being 150 mg shall be determined for one detector per array, and three representative weights, one weight within 0-30 mg, one weight within 31-60 mg, and one weight within 61-100 mg, must agree for each detector in the given array at the 95% confidence level.
- 2.5 The laboratory shall generate and submit self-absorption curves a minimum of every 3 years for gross alpha and gross beta analyses using NIST-traceable standards, or equivalent standard reference materials, in which at least 10,000 counts are accumulated for each planchet count for ^{137}Cs , ^{90}Sr , and ^{241}Am with the maximum weights used for calibration curves being 150 mg. These curves and associated raw data shall be submitted at initiation of the contract and as generated. Planchets older than three years are not acceptable.

3. Continuing Calibrations

- 3.1 The laboratory shall perform weekly continuing calibration verification analyses. The laboratory shall perform standard statistical tests as referenced in the Glossary to determine the instrument reliability and shall maintain this information in logbooks or on benchsheets for on-site technical audits and submit this data monthly or on an as requested basis. This data shall include: daily or weekly reliability checksource name; certification date, expiration date, and DPM activity of standards; count durations; alpha and beta counts obtained; efficiency obtained on the daily or weekly standard; and action taken if the instrument is outside statistical criteria.
- 3.2 Sample analysis shall begin on instruments which have had comprehensive maintenance or have been out of service only after the instrument has passed at least 48 hours of instrument performance checks including reliability checksource and instrument background counts.

- 3.3 The laboratory shall check the plateau(s) or response(s) to the checksource(s) after each gas change and verify that the stability of the instrument remains constant.
- 3.4 Instrument background counts shall be taken a minimum of weekly, and shall be included with each SDG. Background counts shall be counted for at least the same count durations as the samples. The laboratory shall submit with each SDG the background count duration.

4. Sample Holding Times

- 4.1 Water samples shall be analyzed within 180 days from date of collection.

5. Case Narratives

- 5.1 Case narratives shall be specific for each SDG as to abnormalities encountered with samples. Reasons shall be given why proper aliquot size was not used, or if RDLs were not met. Matrix problems, poor counting precision or poor accuracy shall be explained. Reanalyses shall be explained and the analytical results that are reported shall be identified with their respective reanalysis data. Deviations from SOPs shall be explained.

II. Quality Control Requirements

The Quality Control Requirements section describes the specifications for analyzing LCSs, replicate analyses, laboratory blanks, determining MDAs, sample aliquot, and self-absorption coefficients.

1. Laboratory Control Samples

- 1.1 LCSs shall be analyzed at a frequency of 5% per batch.
- 1.2 The LCS activity shall be from greater than 5x to less than or equal to 30x the RDL.
- 1.3 LCSs shall be prepared and analyzed in the same manner as the samples.
- 1.4 LCSs shall have the same aliquot size as the samples. Using the statistical counting error, the observed LCS value shall be within 3σ control limits of the expected LCS value and shall have a relative percent error not to exceed 22% for Gross Alpha and 15% for Gross Beta analysis.
- 1.6 LCSs shall be counted for the same count durations as the samples.
- 1.7 LCS data shall be submitted with each data package with the expected values.

2. Replicate Analyses

- 2.1 Replicate analyses shall be analyzed at a frequency of 10% per batch.
- 2.2 Replicate samples shall be prepared and analyzed in the same manner as the samples.
- 2.3 Replicate samples shall have the same aliquot size as the samples.
- 2.4 Replicate samples shall have the same RDLs as the samples.
- 2.5 Replicates shall be counted for the same count durations as the samples.
- 2.6 Replicate analyses data shall be submitted with each SDG.
- 2.7 The replicate analyses shall be within the 3σ range of the weighted average and its associated standard error. See the Glossary for the Weighted Average formula.

3. Laboratory blanks

- 3.1 Laboratory blanks shall be analyzed at a frequency of 5% per batch.
- 3.2 Laboratory blanks shall be prepared and analyzed in the same manner as the samples. The laboratory may use a deionized water matrix for laboratory soil blanks.
- 3.3 Laboratory blanks shall have the same aliquot size as the samples.
- 3.4 Laboratory blanks shall be counted for the same count duration as the samples.

4. Minimum Detectable Activities

- 4.1 Count durations for samples, replicates, blanks, and backgrounds shall be optimized so that the MDAs achieve the RDLs. Interferences, high dissolved solids, and other matrix problems may cause the sample MDAs to exceed the desired MDAs; however, the laboratory shall demonstrate (usually by reanalysis) that the MDA could not be met due to the matrix and not because of inadequate count time, laboratory problems, or other limitations. Reanalysis due to demonstrated matrix problems will be treated as an additional sample analysis. In all cases, failure to achieve the required MDAs shall be fully explained in the Case Narratives.
- 4.2 The MDAs shall be reported on the sample calculations sheet. The last background count taken (1 month old or less) shall be used for calculations.

III. Raw Data and Documentation

The Raw Data and Documentation requirements section describes the specifications for reporting calculations, reporting raw data from counters, and calculating MDAs.

1. Calculation Sheets

- 1.1 Gross Alpha-Beta calculation sheets shall include: Batch numbers, sample IDs, alpha crosstalk values (if applicable), beta efficiency curve coefficients, beta crosstalk values (if applicable); sample and background count durations, alpha background counts, beta background counts, LCS IDs, observed alpha and beta LCS and expected LCS values, count dates, aliquot volumes, weights on planchets, alpha counts, beta counts, alpha and beta sample activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.16).
- 1.2 A summary report data section shall include: batch numbers, sample IDs, sample collection dates, activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.16).

2. Data from the Counter

- 2.1 The laboratory shall submit raw data; instrument program printouts which include: detector ID numbers, sample IDs, count dates, sample and background count durations, detector identification, and alpha and beta accumulated sample and background counts.

3. Calculating MDAs

- 3.1 The MDA shall be calculated as follows:

$$\text{MDA (pCi/L)} = \frac{4.66 (\text{BKG}/T_1)^{0.5}}{\text{Eff} * \text{Aliquot} * 2.22 * e^{-\lambda t}}$$

Where BKG = Background count rate in CPM
 T_1 = Sample count duration
 Eff = Detector efficiency
 Aliquot = Aliquot in appropriate units (see Scope of Work Sec. B.2.16)
 $e^{-\lambda t}$ = Decay correction (for specific radionuclides)

4. Equipment

- 4.1 The laboratory shall calibrate non-class A pipets on a quarterly basis to ensure that standard aliquots are delivered. The calibration data shall be available on an as requested basis and be retained by the laboratory for on-site technical audits.

REQUIREMENTS FOR $^{226}\text{Radium}$ ANALYSES BY RADON EMANATION

EG&G Rocky Flats
Environmental Management Department

The following are requirements for $^{226}\text{Radium}$ by Radon Emanation analyses. These requirements address instrumentation, calibrations, sample holding times, case narratives, quality control including LCSs, replicate analyses, laboratory blanks, MDAs, cell constants, documentation for calculations, and raw data from counters.

1. General Requirements

The General Requirements section describes the specifications for instrumentation, initial and continuing calibrations, sample holding times, and Case Narratives.

1. Instrumentation

- 1.1 The alpha scintillation detection system should consist of a photomultiplier tube with a light-tight metal housing used in combination with Lucas cells and having a known efficiency and background.
- 1.2 The laboratory shall identify the instrument manufacturer, model, instrument configuration, program and instrument settings, voltages, dates of installation, and cell/instrument identifications (if applicable).
- 1.3 The laboratory shall set up instrumentation according to the manufacturer's instructions; any changes or modifications thereto shall be documented in the instrument maintenance log and maintained for on-site technical audits.
- 1.4 Counter efficiency, aliquot size, and count duration shall be optimized so that the MDAs meet the RDLs for the samples.
- 1.5 The instrumentation shall be surge protected.

2. Initial Calibration

- 2.1 The laboratory should follow instrument manufacturer's instructions using appropriate sources to set voltages and calibrate instruments.
- 2.2 The laboratory shall submit calibration verification data annually or whenever recalibrations are performed. Calibration verification data shall include: identification of each cell/instrument combination; certification date, expiration date, and DPM activities of standards used to prepare the high disk standard; counts obtained and count durations for the high disk standard; calibration dates; and "Midpoint" voltage of the plateau curve for each photomultiplier tube.

- 2.3 Calibrations shall be performed using a high and low disk standard prepared from NIST-traceable standard reference materials, or equivalent standard reference materials, in which at least 10,000 counts are accumulated for $^{226}\text{Radium}$ for the high disk standard.
- 2.4 The laboratory shall perform daily continuing calibration verification analyses with each SDG using a low disk standard (approximately 10 CPM). The laboratory shall perform standard statistical tests as referenced in the Glossary to determine the instrument reliability and shall maintain this information in logbooks or on benchsheets for on-site technical audits and submit this data with each SDG. This data shall include: identification of each cell/instrument combination; identification certification date, expiration date, and DPM activity of standard used to prepare the low disk standard; counts obtained and count durations for the low disk standard; and action taken if the instrument is outside statistical criteria.
- 2.5 Sample analysis shall begin on instruments which have had comprehensive maintenance or have been out of service only after the instrument has passed at least 48 hours of instrument performance checks including reliability checksource counts.
- 2.6 Cell background counts shall be taken a minimum of weekly or before each batch, and shall be included in the appropriate data packages with background counts counted for at least the same count durations as the samples. Cells used for analysis of EG&G samples shall not have backgrounds which exceed 0.15 CPM.

3. Cell Constants

- 3.1 The laboratory shall determine the efficiency of each cell/instrument combination a minimum of annually. The efficiency data shall be submitted on a one time basis or as requested. This data shall include: identification of each cell/instrument combination, concentration in pCi/L of solutions in the "standard bubblers", time interval between initial and final deemanations, time interval between final deemanation and beginning of count, and counts obtained and count durations.

4. Sample Holding Times

- 4.1 Water samples shall be analyzed within 180 days from date of collection.

5. Case Narratives

- 5.1 Case narratives shall be specific for each SDG as to abnormalities encountered with samples. Reasons shall be given why proper aliquot size was not used, or if RDLs were not met. Matrix problems, poor counting precision or poor accuracy shall be explained. Reanalyses shall be explained and the analytical results that are reported shall be identified with their respective reanalysis data. Deviations from SOPs shall be explained.

II. Quality Control Requirements

The Quality Control Requirements section describes the specifications for analyzing LCSs, replicate analyses, laboratory blanks, and determining MDAs, and sample aliquots.

1. Laboratory Control Samples

- 1.1 LCSs shall be analyzed at a frequency of 5% per batch.
- 1.2 The LCS activity shall be from greater than 5x to less than or equal to 30x the RDL.
- 1.3 LCSs shall be prepared and analyzed in the same manner as the samples.
- 1.4 LCSs shall have the same aliquot size as the samples.
- 1.5 Using the statistical counting error, the observed LCS value shall be within 3σ control limits of the expected LCS value and shall have a relative percent error, not to exceed 10%.
- 1.6 LCSs shall be counted for the same count durations as the samples.
- 1.7 LCS data shall be submitted with each data package with the expected values.

2. Replicate Analyses

- 2.1 Replicate analyses shall be analyzed at a frequency of 10% per batch.
- 2.2 Replicate samples shall be prepared and analyzed in the same manner as the samples.
- 2.3 Replicate samples shall have the same aliquot size as the samples.
- 2.4 Replicate samples shall have the same RDLs as the samples.
- 2.5 Replicates shall be counted for the same count durations as the samples.
- 2.6 Replicate analyses data shall be submitted with each data package.
- 2.7 The replicate analyses shall be within the 3σ range of the weighted average and its associated standard error.

3. Laboratory blanks

- 3.1 Laboratory blanks shall be analyzed at a frequency of 5% per batch.
- 3.2 Laboratory blanks shall be prepared and analyzed in the same manner as the samples.
- 3.3 Laboratory blanks shall have the same aliquot size as the samples.
- 3.4 Laboratory blanks shall be counted for the same count duration as the samples.

4. Minimum Detectable Activities

- 4.1 Count durations for samples, replicates, blanks, and backgrounds shall be optimized so that the MDAs achieve the RDLs. Interferences, high dissolved solids, and other matrix problems may cause the sample MDAs to exceed the desired MDAs; however, the laboratory shall demonstrate (usually by reanalysis) that the MDA could not be met due to the matrix and not because of inadequate count time, laboratory problems, or other limitations. Reanalysis due to demonstrated matrix problems will be treated as an additional sample analysis. In all cases, failure to achieve the required MDAs shall be fully explained in the Case Narratives.
- 4.2 The MDAs shall be reported on the sample calculations sheet. The last cell background count taken (1 week old or less) shall be used for calculations.

III. Raw Data and Documentation

The Raw Data and Documentation requirements section describes the specifications for reporting calculations, reporting raw data from counters, and calculating MDAs.

1. Calculation Sheets

- 1.1 ²²⁶Radium by Radon emanation analysis calculation sheets shall include: Batch numbers, sample IDs, identification of each cell/instrument combination, cell constant for each cell/instrument combination, recovery factors (if applicable); sample and background count durations, sample and background counts, time interval between initial and final deemanation and beginning of count, LCS IDs, observed LCS activities in pCi/L with uncertainties and expected LCS values, count dates, aliquot volumes, and sample activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.16).
- 1.2 A summary report data section shall include: batch numbers, sample IDs, sample collection dates, activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.16).

2. Data from the Counter

- 2.1 The laboratory shall submit raw data and instrument printouts which include: sample IDs, count dates, sample and background count durations, identification of cell/instrument combination, and accumulated sample and background counts.

3. Calculating MDAs

3.1 The MDA shall be calculated as follows:

$$\text{MDA (pCi/L for water)} = \frac{4.66}{\text{Eff} * \text{Vol} * 2.22} \left(\frac{\text{BKG/T}_1}{1 - e^{-\lambda_1}} \right)^{0.5} * \frac{1}{e^{-\lambda_2}} * \frac{1}{1 - e^{-\lambda_3}}$$

Where BKG = Background count rate in CPM

T_1 = Sample count duration

Eff = Detector efficiency

Vol = Volume in Liters

$1 - e^{-\lambda_1}$ = Ingrowth correction for Radon²²² from initial to final deaeration

$e^{-\lambda_2}$ = Decay correction for Radon²²² from final deaeration to beginning of count

$e^{-\lambda_3}$ = Decay correction for Radon²²² during the count

4. Equipment

4.1 The laboratory shall calibrate on a quarterly basis, non-class A pipets and sample dispensers to ensure that standard aliquots are delivered. The data shall be available on an as requested basis and be retained by the laboratory for on-site audits.

REQUIREMENTS FOR RADIOMETRIC STRONTIUM, CESIUM AND ²²⁸RADIUM ANALYSES BY GAS PROPORTIONAL COUNTING

EG&G Rocky Flats
Environmental Management Department

The following are requirements for Radiometric Strontium, Cesium and ²²⁸Radium analyses using GPC. These requirements address instrumentation, calibrations, sample holding times, case narratives, quality control including lab control samples, replicate analyses, laboratory blanks, minimum detectable activities, chemical recovery factors, efficiency factors, documentation for calculations, and raw data from counters.

I. General Requirements

The General Requirements section describes the specifications for instrumentation, initial and continuing calibrations, sample holding times, and Case Narratives.

1. Instrumentation

- 1.1 Instrumentation shall consist of any low-background, anti-coincidence proportional counter consisting of : sample detector, cosmic detector, and pulse height discriminating circuitry for measuring beta activity, or demonstrated equivalent.
- 1.2 The laboratory shall identify the instrument manufacturer, model, instrument configuration, program and instrument settings, voltages, dates of installation, and detector identifications.
- 1.3 The laboratory shall set up instrumentation according to the manufacturer's instructions; any changes or modifications thereto shall be documented.
- 1.4 Counter efficiency, aliquot size, and count duration shall be optimized so that the MDAs meet the RDLs for the samples.
- 1.5 The instrument shall be surge protected.

2. Initial Calibration

- 2.1 The laboratory should follow instrument manufacturer's instructions using appropriate sources to set voltages and calibrate instrument detectors.

- 2.2 The laboratory shall submit calibration verification data every three years or whenever re-calibrations are performed. Calibration verification data shall include: radionuclide name; certification, expiration dates, and DPM activities of standards; volumes of standards used; count durations; calibration dates; mg weights of salts; beta counts obtained; efficiencies; and best-fit curve coefficients.
- 2.3 The laboratory shall perform calibrations for each radionuclide to be counted, and the standard reference material shall have the same physical form (size, shape, planchet material, etc.) as the samples to be counted.
- 2.4 For multiple counting systems, calibration equivalency shall be shown for each detector in the array. The complete self-absorption curve shall be determined for one detector per array, and three representative weights, one weight within 0-30 mg, one weight within 31-60 mg, and one weight within 61-125 mg. The weights must agree within 3σ control limits using the referenced statistical tests (see the Glossary).
- 2.5 The laboratory shall generate and submit at least every three years self-absorption curves for $^{90}\text{Strontium}$, $^{137}\text{Cesium}$, and $^{228}\text{Actinium}$ or comparable beta energy nuclide such as $^{89}\text{Strontium}$. The curves shall be prepared using NIST-traceable standards, or equivalent standard reference materials, accumulating at least 10,000 counts for each planchet counted for $^{90}\text{Strontium}$, $^{137}\text{Cesium}$ and $^{228}\text{Actinium}$ or comparable beta energy nuclide such as $^{89}\text{Strontium}$. These curves and associated raw data shall be submitted at initiation of contract and as generated. Planchets older than three years are not acceptable.

3. Continuing Calibrations

- 3.1 The laboratory shall perform weekly continuing calibration verification analyses. The laboratory shall perform standard statistical tests referenced in the Glossary to determine the instrument reliability and submit this data monthly or on an as requested basis. The laboratory shall maintain this information in logbooks or on benchsheets for on-site technical audits. This data shall include: daily or weekly reliability checksource name; certification date, expiration date, and DPM activity of standards; count durations; beta counts obtained; efficiency obtained on the daily or weekly standard; and action taken if the instrument is outside statistical criteria.
- 3.2 Sample analysis shall begin on instruments which have had comprehensive maintenance or have been out of service only after the instrument has passed at least 48 hours of instrument performance checks including reliability check source and instrument background counts.

- 3.3 The laboratory shall check the plateau(s) or response(s) to the checksource(s) after each gas change and verify that the stability of the instrument remains constant.
- 3.4 Instrument background counts shall be taken a minimum of weekly, or before each batch, and shall be included in each SDG. Background counts shall be counted for at least the same count durations as the samples. The laboratory shall submit with each SDG the background count duration.

4. Sample Holding Times

- 4.1 Water samples shall be analyzed within 180 days from date of collection.

5. Case Narratives

- 5.1 Case narratives shall be specific for each SDG as to abnormalities encountered with samples. Reasons shall be given why proper aliquot size was not used, or if RDLs were not met. Matrix problems, poor counting precision or poor accuracy shall be explained. Reanalysis shall be explained and the analytical results that are reported shall be identified with their respective reanalysis data. Deviations from SOPs shall be explained.

II. Quality Control Requirements

The Quality Control Requirements section describes the specifications for analyzing LCSs, replicate analyses, laboratory blanks, and determining MDAs, sample aliquot, and self-absorption coefficients.

1. Laboratory Control Samples

- 1.1 LCSs shall be analyzed at a frequency of 5% per batch.
- 1.2 The LCS activity shall be from greater than 5x or equal to 30x the instrument RDL.
- 1.3 LCSs shall be prepared and analyzed in the same manner as the samples.
- 1.4 LCSs shall have the same aliquot size as the samples.
- 1.5 Using the statistical counting error, the observed LCS value shall be within 3 σ control limits of the expected value and shall have a relative percent error not to exceed 25% for total Radiostrontium and Cesium and 30% for ²²⁸Radium.

- 1.6 LCSs shall be counted for the same count durations as the samples.
- 1.7 LCS data shall be submitted with each data package with the expected values.

2. Replicate Analyses

- 2.1 Replicate analyses shall be analyzed at a frequency of 10% per batch.
- 2.2 Replicate samples shall be prepared and analyzed in the same manner as the samples.
- 2.3 Replicate samples shall have the same aliquot size as the samples.
- 2.4 Replicate samples shall have the same RDLs as the samples.
- 2.5 Replicates shall be counted for the same count durations as the samples.
- 2.6 Replicate analyses data shall be submitted with each data package.
- 2.7 The replicate analyses shall be within the 3σ range of the weighted average and its associated standard error. See the Glossary for the Weighted Average formula.

3. Laboratory blanks

- 3.1 Laboratory blanks shall be analyzed at a frequency of 5% per batch.
- 3.2 Laboratory blanks shall be prepared and analyzed in the same manner as the samples. The laboratory may use a deionized water matrix for laboratory soil blanks.
- 3.3 Laboratory blanks shall have the same aliquot size as the samples.
- 3.4 Laboratory blanks may be counted for the same count duration as the samples.

4. Minimum Detectable Activities

- 4.1 Count durations for samples, replicates, blanks, and backgrounds shall be optimized so that the MDAs achieve the RDLs. Interferences, high dissolved solids, and other matrix problems may cause the sample MDAs to exceed the desired MDAs; however, the laboratory shall demonstrate (usually by reanalysis) that the MDA could not be met due to the matrix and not because of inadequate count time, laboratory problems, or other limitations. Reanalysis due to demonstrated matrix problems will be treated as an additional sample analysis. In all cases, failure to achieve the required MDAs shall be fully explained in the Case Narratives.

- 4.2 The MDAs shall be reported on the sample calculations sheet. The last background count taken (1 week old or less) shall be used for calculations.

6. Chemical Recovery Factors

- 6.1 The chemical recovery factor is used to calculate the sample activity, uncertainty and MDA. The laboratory shall submit with each SDG the amount of standardized stable carrier(s) or radionuclide carrier used in the procedure to determine the chemical recovery.
- 6.2 The chemical recovery for the procedures shall be greater than 35% but less than 100%. Chemical recoveries greater than 100% are not acceptable.

III. Raw Data and Documentation

The Raw Data and Documentation requirements section describes the specifications for reporting calculations, reporting raw data from counters, and calculating MDAs.

1. Calculation Sheets

- 1.1 Radiometric calculation sheets shall include: Batch numbers, sample IDs, beta efficiency curve coefficients, sample and background count durations, beta background counts, LCS IDs, observed beta LCS and expected LCS values, count dates, aliquot volumes, weights, amount of standardized carrier added, beta counts, beta sample activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.16).
- 1.2 A summary report data section shall include: batch numbers sample IDs, sample collection dates, activities, uncertainties, and MDAs in appropriate units (see Scope of Work Section B.2.16).

2. Data from the Counter

- 2.1 The laboratory shall submit raw data; instrument program printouts, which include: sample IDs, count dates, sample and background count durations, detector identification, and accumulated beta sample and background counts.

3. Calculating MDAs

3.1 The MDA shall be calculated as follows:

MDA (pCi/Aliquot in appropriate units (see section B.2.16)

$$= \frac{4.66 (BKG/T_1)^{0.5}}{\text{Eff} * \text{Aliquot} * 2.22 * e^{-\lambda t}}$$

Where BKG = Background count rate in CPM
 T_1 = Sample count duration
 Eff = Detector efficiency
 Aliquot = Aliquot in appropriate units (see section B.2.16)
 $e^{-\lambda t}$ = Decay correction (for specific radionuclides)

4. Equipment

4.1 The laboratory shall calibrate on a quarterly basis, non-class A pipets and sample dispensers to ensure that standard aliquots are delivered. The data shall be available on an as requested basis and be retained by the laboratory for on-site audits.

EXHIBIT II

RADIOCHEMISTRY DATA PACKAGE CHECKLISTS

**Radiochemistry Data Completeness
Checklist for Alpha Spectrometric Analyses**

- A. ☐ Case Narrative
 ☐ Batch number
 ☐ Abnormalities, reanalyses, or SOP deviations explained
 ☐ Matrix problems explained
 ☐ Instrument problems explained
 ☐ Improper collection, storage, preservation, container explained
 ☐ Hold times or RDLs met, explained if not met
- B. ☐ Efficiency, Background, and Calibrations Data Package
 ☐ ID of each Detector
 ☐ Dates of last efficiency check including: spectra and/or channel
 by channel printout, certificates and DPMs of check sources;
 counts obtained; count durations; and channels selected for ROIs
 ☐ Proper channel numbers of isotopes of interest, based on
 calibration data of Pu, Am, Cm, and U standards
 ☐ Total memory (channels per detector)
 ☐ Energy range of the alpha detection system (KeV)
 ☐ Gain (KeV/channel) of the alpha detection system
 ☐ Dates of last background spectra including: spectra and/or channel
 by channel printout; count durations; counts obtained; and channels
 selected for ROIs
- C. ☐ Laboratory Blanks Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date laboratory blanks were analyzed
 ☐ ID of samples analyzed with the laboratory blanks
 ☐ Type of method blank used, MDA of method
 ☐ Volume of aliquot for laboratory blanks
- D. ☐ Replicate Sample Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date sample and replicates were analyzed
 ☐ Sample IDs, values obtained for sample and replicates
 ☐ Count durations of sample and replicates
 ☐ Volume of aliquot for sample and replicates
 ☐ Calculated uncertainties and MDAs
- E. ☐ Lab Control Samples (LCSs) Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date LCSs were analyzed
 ☐ ID of LCS, ID of spike concentrate used to prepare LCS,
 and expected value with uncertainty
 ☐ Values obtained for LCSs with uncertainty and MDA
 ☐ ID of samples analyzed with the LCSs

- F. ☐ Resolution
 ☐ System gain (in KeV/Channel)
 ☐ FWHM (in channels or KeVs)
 ☐ Counts in peak channel for LCS
- G. ☐ Recovery Factors
 ☐ Efficiency factor provided for each detector used
 ☐ ID of each detector used
 ☐ Net counts and FWHM obtained for each isotopic tracer used
 ☐ Count duration
 ☐ ID, FWHM, and DPM value of each isotopic tracer
 ☐ Calculated chemical recovery
- H. ☐ Sample Data Package
 ☐ Copy of Chain of Custody (COC)
 ☐ Printed report of results for samples and reruns
 ☐ Computer calculations sheet including: sample IDs,
 detector IDs, ROIs for isotopes of interest, counts obtained for
 samples, background counts obtained, ROIs for tracer, isotopic
 tracer counts obtained count durations, DPMs of tracer used,
 aliquots of samples and tracers, detector efficiency, chemical
 recoveries, activities, uncertainties, and MDAs obtained for samples
- I. ☐ Minimum Detectable Activity
 ☐ Background measurements including: counts and
 count durations of samples and backgrounds taken during
 the same weekly time period
 ☐ Date of analysis
 ☐ Background CPM
 ☐ MDA calculated for each isotopic analysis for the sample

Radiochemistry Data Completeness
Checklist for Tritium Analyses

- A. ☐ Case Narrative
 ☐ Batch number
 ☐ Abnormalities, reanalyses, or SOP deviations explained
 ☐ Matrix problems explained
 ☐ Instrument problems explained
 ☐ Improper collection, storage, preservation, container explained
 ☐ Hold times or RDLs met, explained if not met
- B. ☐ Initial and Continuing Calibration Data Package
 ☐ Instrument ID number, manufacturer, and model number, with program settings
 ☐ Date of performance check
 ☐ NIST-traceable reference material certificates with expiration date and DPM activity of unquenched standard
 ☐ Raw data from counter showing counts obtained and count durations
 ☐ Efficiency obtained for unquenched standard
 ☐ Quench monitor values and CPM for Standard used to check long term performance of cocktail and instrument
 ☐ Background water and preparation blank vials CPM results
- C. ☐ Background Water and Preparation Blanks Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date background water and preparation blanks were analyzed
 ☐ ID of samples analyzed with the background water and preparation blanks
 ☐ Type of method blank used, MDA of method
 ☐ Volume of aliquot for background water and preparation blanks
- D. ☐ Replicate Sample Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date sample and replicates were analyzed
 ☐ Collection date
 ☐ Sample IDs, values obtained for sample and replicates
 ☐ Count durations of sample and replicates
 ☐ Volume of aliquot for sample and replicates
 ☐ Calculated uncertainties and MDAs
- E. ☐ Lab Control Samples (LCSs) Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date LCSs were analyzed
 ☐ ID of LCS, ID of spike concentrate used to prepare LCS, and expected value with uncertainty
 ☐ Values obtained for LCSs with uncertainty and MDA
 ☐ ID of samples analyzed with the LCSs

- F. ☐ Minimum Detectable Activity
☐ Background measurements including: counts and count durations of samples and backgrounds taken during the same weekly time period
☐ Date of analysis
☐ Background CPM
☐ MDA calculated for each isotopic analysis for the sample
- G. ☐ Quench and Efficiency
☐ Detector ID
☐ NIST-traceable reference material certificates used to determine detector efficiencies and quench curve
☐ Quench monitor used
☐ Quench monitor values and efficiency values
☐ Date of last quench curve check
☐ Batch number and sample IDs
☐ Efficiency standard and backgrounds used
☐ Volume added to cocktail, cocktail used, and vials used
☐ Instrument settings used when detector efficiencies and quench curve were analyzed
☐ Volume of spike added to cocktail for internal standardization method
☐ Best fit curve coefficients for quench curve method
- H. ☐ Sample Data Package
☐ Copy of Chain of Custody
☐ Printed report of results for sample, reruns
☐ Computer calculations sheet including: sample IDs, isotopes of interest, counts obtained for samples, background counts obtained, sample aliquot, collection date, count date, count duration, detector efficiency, and activities obtained for samples, uncertainties, and MDAs
☐ Raw data from counter, copies of notebook pages
☐ Instrument counting logs

Radiochemical Data Completeness
Checklist for Gamma Spectrometric Analyses

- A. ☐ Case Narrative
- ☐ Batch Number
 - ☐ Abnormalities, reanalyses, or SOP deviations explained
 - ☐ Matrix problems explained
 - ☐ Instrument problems explained
 - ☐ Improper collection, storage, preservation, container explained
 - ☐ Hold times or RDLs met, explained if not met
- B. ☐ Calibrations Data Package
- ☐ ID of each Detector
 - ☐ Date of the calibration check; channel by channel printout, identification, certification and expiration dates, and DPS values of checksources; count durations; calibrated energy (in KeV) for each peak of interest; calibrated centroid channel number for each peak of interest; observed channel number for each peak of interest; offset value, and calculated slope from the least squares fit of the calibration data
 - ☐ Full Width Half Maximum (FWHM) of the peaks
 - ☐ Energy range of the gamma detection system in (KeV); channels of memory
 - ☐ Geometry, matrix, weight for which the efficiency curve is constructed
 - ☐ line intensity of each nuclide of interest; counts per second observed for each peak of interest; DPS value of each nuclide; observed efficiency, observed energy, observed channel number of each nuclide; and plot of energy versus efficiency
 - ☐ Integrated area of the peak ROIs; count duration
 - ☐ Dates of last background spectra including: spectra and/or channel by channel printout; count durations; counts obtained for the peak ROIs; and compared to a long term background spectra
- C. ☐ Matrix Blanks Data Package
- ☐ ID of each detector used
 - ☐ Identification of the blank (i.e. if Ottawa Sand for Soil, etc) or deionized water
 - ☐ Integrated area of peaks ROIs
 - ☐ Count duration
 - ☐ Date matrix blanks were analyzed
 - ☐ ID of samples analyzed with the matrix blanks
 - ☐ Type of method blank used, MDA of method
 - ☐ Volume of aliquot, weight, matrix, and geometry for matrix blanks

- D. ☐ LCSs Data Package
- ☐ ID of each detector used
 - ☐ Analyst initials
 - ☐ Date LCSs were analyzed
 - ☐ ID, aliquot size, weight, and geometry of LCS
 - ☐ Integrated areas of LCS peaks
 - ☐ Background counts
 - ☐ Count duration
 - ☐ Values obtained for LCSs with uncertainty and MDA
 - ☐ Expected value of LCSs with uncertainty
 - ☐ ID of samples analyzed with the LCSs
 - ☐ Results of statistical evaluation for accuracy
- E. ☐ Quality Control for Gamma Analysis Data Package
- ☐ Detector identification number
 - ☐ NIST traceable certificate and DPM value of source
 - ☐ Calculated activities of a nuclide of interest based on independent analysis of multiple lines
 - ☐ Latest background spectra (channel by channel)
 - ☐ Results of the latest background spectra
 - ☐ FWHM of the Co⁶⁰ peak at 1332 KeV
 - ☐ Net counts per second obtained for each nuclide of interest in the calibration
 - ☐ Observed efficiency (%) of each nuclide of interest
 - ☐ Observed energy and channel number of nuclides of interest, in addition to calibrated energies and channel numbers
 - ☐ Activities of nuclides of interest used for energy calibration
- F. ☐ Sample Data Package
- ☐ Copy of Chain of Custody
 - ☐ Printed report of results for samples and reruns
 - ☐ Computer calculations sheet including: detector identification number; date of analysis; sample number; names of nuclides detected; count duration; energy and channel number for each analysis; integrated area for each peak ROI, FWHM of each peak of interest; peak width for each ROI; calculated counts per second for each nuclide of interest; weight, matrix, and geometry of the samples; and calculated activity and uncertainty of the samples
- G. ☐ Minimum Detectable Activity
- ☐ Background spectra for each detector showing background counts accumulated for each nuclide of interest
 - ☐ Count duration for background
 - ☐ Date of analysis
 - ☐ Background CPM
 - ☐ MDA calculated for each nuclide of interest for the sample

Radiochemical Data Completeness
Checklist for Gross α & β Analyses
by Gas Proportional Counters

- A. ☐ Case Narrative
- ☐ Batch Number
 - ☐ Abnormalities, reanalyses, or SOP deviations explained
 - ☐ Matrix problems explained
 - ☐ Instrument problems explained
 - ☐ Improper collection, storage, preservation, container explained
 - ☐ Hold times or RDLs met, explained if not met
- B. ☐ Initial and Continuing Calibration Data Package
- ☐ Detector ID
 - ☐ Date and time calibrated, calibration check, analyst initials
 - ☐ Radionuclide standard name, NIST certification and expiration dates, and DPM value
 - ☐ Aliquots of standards used
 - ☐ Raw data from counters showing alpha and beta counts obtained and count durations for each weight of salt
 - ☐ Weights of salts
 - ☐ Efficiencies
 - ☐ Alpha/Beta crosstalk values
 - ☐ Best fit curve coefficients
 - ☐ Reliability checksource name, NIST certification, expiration and DPM activity
 - ☐ Raw data from counters showing alpha and beta counts obtained and count durations for reliability checksource
 - ☐ Efficiency obtained for checksource
 - ☐ Background counts obtained and count duration for each detector
- C. ☐ Laboratory Blanks Data Package
- ☐ ID of each detector used
 - ☐ Analyst initials
 - ☐ Date laboratory blanks were analyzed
 - ☐ ID of samples analyzed with the laboratory blanks
 - ☐ Type of method blank used, MDA of method
 - ☐ Volume of aliquot for laboratory blanks
- D. ☐ Replicate Sample Data Package
- ☐ ID of each detector used
 - ☐ Analyst initials
 - ☐ Date sample and replicates were analyzed
 - ☐ Sample IDs, values obtained for sample and replicates
 - ☐ Count duration of sample and replicates
 - ☐ Volume of aliquot for sample and replicates
 - ☐ Calculated uncertainties and MDAs

- E. ☐ LCSs Data Package
☐ ID of each detector used
☐ Analyst initials
☐ Date LCSs were analyzed
☐ ID of LCS; ID of spike concentrate used to prepare LCS,
and expected value with uncertainty
☐ Values obtained for LCSs with uncertainty and MDA
☐ ID of samples analyzed with the LCSs
- F. ☐ MDA
☐ Background measurements including: counts and
count durations of samples and backgrounds taken during
the same weekly time period
☐ Date of analysis
☐ Background CPM
☐ MDA calculated for both gross alpha and gross beta
analysis of the sample
- G. ☐ Size of Aliquot in Gross α & β Determination Data Package
☐ Sample ID
☐ Volume of sample to deliver solids on the planchet
☐ Raw data supporting efficiency factor and efficiency factor used
- H. ☐ Sample Data Package
☐ Copy of Chain of Custody
☐ Printed report of results for sample, reruns
☐ Raw data from counter, copies of notebook pages
☐ Manual/Computer calculations
☐ Sample ID, Detector ID, obtained sample and background
counts and count durations observed, aliquot of sample,
weight of solids counted, detector efficiency, activities,
uncertainties, and MDAs

Radiochemical Data Completeness
Checklist ²²⁶Radium Analysis by Radon
Emanation of Water

- A. ☐ Case Narrative
 ☐ Batch Number
 ☐ Abnormalities, reanalyses, or SOP deviations explained
 ☐ Matrix problems explained
 ☐ Instrument problems explained
 ☐ Improper collection, storage, preservation, container explained
 ☐ Hold times or RDLs met, explained if not met
- B. ☐ Initial and Continuing Calibration Data Package
 ☐ ID of each detector
 ☐ Calibration dates for each detector
 ☐ Identification, certification dates, expiration date and DPM values
 of the standard reference material used to prepare high and
 low disk standards
 ☐ DPM values of the high and low disk standards
 ☐ Raw data from counters showing counts obtained and count duration
 for high and low disk standards
 ☐ Midpoint voltage of the plateau curve for the photomultiplier
 tube
 ☐ Statistical analysis of the weekly checksources
 ☐ Background counts obtained for each Lucas cell
 with count durations
- C. ☐ Laboratory Blanks Data Package
 ☐ ID of each cell/instrument combination used
 ☐ Analyst initials
 ☐ Date laboratory blanks were analyzed
 ☐ ID of samples analyzed with the laboratory blanks
 ☐ Type of method blank used, MDA of method
 ☐ Volume of aliquot for laboratory blanks
- D. ☐ Replicate Sample Data Package
 ☐ ID of each cell/instrument combination used
 ☐ Analyst initials
 ☐ Date sample and replicates were analyzed
 ☐ Values obtained for sample and replicates
 ☐ Count durations of samples and replicates
 ☐ Volume of aliquot for sample and replicates
 ☐ Calculated uncertainties and MDAs

- E. ☐ LCSs Data Package
- ☐ ID of each cell/instrument combination used
 - ☐ Analyst initials
 - ☐ Date LCSs were analyzed
 - ☐ ID of LCS, ID of spike concentrate used to prepare LCS, and expected value with uncertainty
 - ☐ Values obtained for LCSs with uncertainty and MDA
 - ☐ ID of samples analyzed with the LCSs
- F. ☐ Cell Constants Data Package
- ☐ ID of each cell/instrument combination used
 - ☐ Concentration in PCi/L of solutions used in "standard bubblers"
 - ☐ Time interval between initial and final deemanations
 - ☐ Counts obtained and count durations of standard for each cell/instrument combination
 - ☐ Efficiency obtained for each cell/instrument combination
 - ☐ Results of statistical evaluation of cell/instrument efficiencies
- G. ☐ Sample Data Package
- ☐ Copy of Chain of Custody (COC)
 - ☐ Printed report of results obtained for samples, lab control samples, replicates, reruns, and laboratory blanks
 - ☐ Computer calculations sheet including:
 - Sample ID, cell/instrument combination identification,
 - counts obtained for sample, counts obtained for background,
 - count durations, sample aliquots used, cell constant values,
 - time intervals between initial and final deemanations, time intervals between final deemanation and counting, calculated sample activity, uncertainty, and MDA
- H. ☐ MDAs Data Package
- ☐ ID of each cell/instrument combination used
 - ☐ Background counts obtained
 - ☐ Sample count duration
 - ☐ Background count duration
 - ☐ Date of analysis
 - ☐ Calculated MDA

Radiochemical Data Completeness
Checklist for Radiometric Strontium, Cesium,
and ²²⁸Radium Analyses by Gas Proportional Counters

- A. ☐ Case Narrative
 ☐ Batch Number
 ☐ Abnormalities, reanalyses, or SOP deviations explained
 ☐ Matrix problems explained
 ☐ Instrument problems explained
 ☐ Improper collection, storage, preservation, container explained
 ☐ Hold times or RDLs met, explained if not met
- B. ☐ Initial and Continuing Calibration Data Package
 ☐ Detector ID
 ☐ Date and time calibrated, calibration check, analyst initials
 ☐ Radionuclide standard name, NIST certification and expiration
 dates, and DPM value
 ☐ Aliquots of standards used
 ☐ Raw data from counters showing beta counts obtained
 and count durations for each weight of salt
 ☐ Weights of salts
 ☐ Efficiencies
 ☐ Best-fit curve coefficients
 ☐ Carrier weights added to planchets
 ☐ Reliability checksource name, NIST certification,
 expiration and DPM activity
 ☐ Raw data from counters showing beta counts obtained
 and count durations for reliability checksource
 ☐ Efficiency obtained for checksource
 ☐ Background counts obtained and count duration for each detector
- C. ☐ Laboratory Blanks Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date laboratory blanks were analyzed
 ☐ ID of samples analyzed with the laboratory blanks
 ☐ Type of method blank used, MDA of method
 ☐ Volume of aliquot for laboratory blanks
- D. ☐ Replicate Sample Data Package
 ☐ ID of each detector used
 ☐ Analyst initials
 ☐ Date sample and replicates were analyzed
 ☐ Sample IDs, values obtained for sample and replicates
 ☐ Count duration of sample and replicates
 ☐ Volume of aliquot for sample and replicates
 ☐ Calculated uncertainties and MDAs

- E. ☐ LCSs Data Package
☐ ID of each detector used
☐ Analyst initials
☐ Date LCSs were analyzed
☐ ID of LCS, ID of spike concentrate used to prepare LCS,
and expected value with uncertainty
☐ Values obtained for LCSs with uncertainty and MDA
☐ ID of samples analyzed with the LCSs
- F. ☐ Chemical Recovery
☐ Volume of aliquot used
☐ Amount of carrier added to the sample
☐ Weight of sample precipitate
☐ Results of Atomic Absorption analysis if the amount
of stable strontium exceeded a few milligrams
☐ Fractional chemical recovery of the beta nuclide of interest
- G. ☐ Efficiency Factors Data Package
☐ Name of beta nuclide of interest
☐ Certification date and DPM value of the standard
☐ Volume of aliquot for standards
☐ Net CPMs obtained for standards
☐ Count date
☐ Efficiency derived from latest self-absorption curve
for the beta nuclide of interest
☐ Best-fit curve coefficients
- H. ☐ Sample Data Package
☐ Copy of Chain of Custody
☐ Printed report of results for sample, reruns
☐ Raw data from counter, copies of notebook pages
☐ Manual/Computer calculations
☐ Sample ID, detector ID, sample and background counts obtained
and count durations, aliquot of samples,
weights of solids counted, detector efficiency, activities,
uncertainties, and MDAs
- I. ☐ MDA
☐ Background measurements including: counts and
count durations of samples and backgrounds taken during
the same weekly time period
☐ Date of analysis
☐ Background CPM
☐ MDA calculated for total Radiostrontium, Cesium, or $^{228}\text{Radium}$
analysis of the sample

EXHIBIT III

CHAIN-OF-CUSTODY AND DOCUMENT CONTROL

SPECIFICATIONS FOR CHAIN-OF-CUSTODY, DOCUMENT CONTROL PROCEDURES, AND WRITTEN STANDARD OPERATING PROCEDURES

1.0 SAMPLE CHAIN-OF-CUSTODY

A sample is physical evidence collected from a facility or from the environment. An essential part of the hazardous waste investigation effort is that the evidence gathered be controlled. To accomplish this, the following sample identification, chain-of-custody, sample receiving, and sample tracking procedures have been established.

1.1 Sample Identification

The contractor shall have a specified method for maintaining identification of samples throughout the laboratory.

Each sample and sample preparation container shall be labeled with the field assigned sample number or a unique laboratory identifier. If a unique laboratory identifier is used, it shall be cross-referenced to the field number.

1.2 Chain-of-Custody Procedures

The contractor shall have procedures ensuring that sample custody is maintained and documented. A sample is under custody if:

- It is in your possession, or
- It is in your view after being in your possession, or
- It was in your possession and you locked it up, or
- It is in a designated secure area. (Secure areas shall be accessible only to authorized personnel.)

1.3 Sample Receiving Procedures

1.3.1 The contractor shall designate a sample custodian responsible for receiving all samples.

1.3.2 The contractor shall designate a representative to receive samples in the event that the sample custodian is not available.

1.3.3 The condition of the shipping containers and sample bottles shall be inspected upon receipt by the sample custodian or his/her representative.

1.3.4 The condition of the custody seals (intact/not intact) shall be inspected upon receipt by the sample custodian or his/her representative.

- 1.3.5 The sample custodian or his/her representative shall check for the presence or absence of the following documents accompanying the sample shipment:
- Airbills
 - Custody seals
 - Custody records
 - Sample tags
- 1.3.6 The sample custodian or his/her representative shall sign and date all forms (e.g., custody records and airbills) accompanying the samples at the time of sample receipt.
- 1.3.7 The sample custodian or his/her representative shall record and cross-reference sample tag identification numbers to the sample label numbers (if not already recorded on the chain-of-custody record(s)).
- 1.3.8 The contractor shall contact the EMAD Radioanalytical Program Chemist or designee to resolve discrepancies and problems such as absent documents, conflicting information, broken custody seals, and unsatisfactory sample condition (e.g., leaking sample bottle).
- 1.3.9 The following information shall be documented by the sample custodian or his/her representative as samples are received and inspected:
- Condition of the shipping container
 - Presence or absence and condition of custody seals on shipping and/or sample containers
 - Condition of the sample bottles
 - Presence or absence of airbills
 - Presence or absence of custody records
 - Presence or absence of sample tags
 - Sample tag identification numbers cross-referenced to the sample label numbers if not recorded on the chain-of-custody record(s)
 - Verification of agreement or non-agreement of information on shipping documents
 - Problems or discrepancies and their resolution

1.4 Sample Tracking Procedures

The contractor shall maintain records documenting all phases of sample handling from receipt to final analysis. The records shall include documentation of the movement of samples and prepared samples into and out of designated laboratory storage areas.

2.0 DOCUMENT CONTROL PROCEDURES

Controlled documents used by contract laboratories shall include, but not be limited to, logbooks, chain-of-custody records, sample work sheets, bench sheets, and other documents relating to the sample or sample analyses. The following document control procedures have been established to assure that all laboratory records are assembled and stored.

2.1 Preprinted Laboratory Forms and Logbooks

- 2.1.1 All documents produced by the subcontractor which are directly related to the preparation and analysis of RFP EM Department samples shall become the property of EG&G. All observations and results recorded by the laboratory but not on preprinted laboratory forms shall be entered into permanent laboratory logbooks. When all data from a sample batch is compiled, all original laboratory forms and copies of all sample-related logbook entries shall be included in the documentation package.
- 2.1.2 The contractor shall identify the activity recorded on all laboratory documents which are directly related to the preparation and analysis of RFP EM Department samples.
- 2.1.3 Pre-printed laboratory forms shall contain the name of the laboratory and be dated and signed by the person responsible for performing the activity at the time an activity is performed.
- 2.1.4 Logbook entries shall be dated and signed by the person responsible for performing the activity at the time an activity is performed.
- 2.1.5 Logbook entries shall be in chronological order. Entries in logbooks, with the exception of instrument run logs and extraction logs, shall include only one sample batch per page.
- 2.1.6 Instrument run logs shall be maintained so as to enable a reconstruction of the analysis and/or counting sequence of individual instruments.

The laboratory shall use only laboratory or field assigned sample identification numbers in the logs for sample identification.
- 2.1.7 Corrections to supporting documents and raw data shall be made by drawing a single line through the error and entering the correct information. Corrections and additions to supporting documents and raw data shall be dated and initialed. No information shall be obliterated or rendered unreadable.

All notations shall be recorded in ink.

Unused portions of documents shall be "z'd" out.

2.2 Consistency of Documentation

The contractor shall assign a document control officer responsible for the organization and assembly of sample files.

Before releasing analytical results, the document control officer shall assemble and cross-check the information on sample tags, custody records, lab bench sheet, personal and instrument logs, and other relevant data to ensure that data pertaining to each particular sample or case is consistent throughout the case file.

2.3 Document Numbering and Inventory Procedure

In order to provide document accountability of the completed analysis records, each item in a case shall be inventoried and assigned a serialized number and sample delivery group identifier.

Sample Delivery Group number - Serialized number (for example: 232-0001)

The number of pages of each item shall be accounted for if each page is not individually numbered. All documents relevant to each case, including logbook pages, bench sheets, spectra, counting logs, self-absorption curves, channel-by-channel printouts, statistical formulas and analyses, custody records, etc., shall be inventoried. The document control officer shall be responsible for ensuring that all documents generated are placed in the file for inventory and are available for inspection. The document control officer shall place the sample tags in plastic bags in the file.

3.0 SPECIFICATIONS FOR WRITTEN STANDARD OPERATING PROCEDURES

The subcontractor shall have written SOPs for receipt of samples, maintenance of custody, sample identification, sample storage, tracking the analysis of samples, and assembly of completed data.

An SOP is defined as a written narrative stepwise description of laboratory operating procedures including examples of laboratory documents. The SOPs should accurately describe the actual procedures used in the laboratory, and copies of the written SOPs shall be available to the appropriate laboratory personnel. The subcontractor's SOPs shall provide mechanisms and documentation to meet each of the following specifications.

- 3.1 The subcontractor shall have written SOPs describing the sample custodian's duties and responsibilities.
- 3.2 The subcontractor shall have written SOPs for receiving and logging in of the samples. The procedures shall include but not be limited to documenting the following information:
 - 3.2.1 Presence or absence of chain-of-custody forms
 - 3.2.2 Presence or absence of airbills
 - 3.2.3 Presence or absence of custody seals on shipping and/or sample containers and their condition
 - 3.2.4 Presence or absence of sample tags
 - 3.2.5 Sample tag ID numbers if not recorded on the chain-of-custody record(s) or packing list(s)
 - 3.2.6 Condition of the shipping container
 - 3.2.7 Condition of the sample bottles
 - 3.2.8 Verification of agreement or non-agreement of information on receiving documents
 - 3.2.9 Resolution of problems or discrepancies
 - 3.2.10 The definition of any terms used to describe sample condition upon receipt
- 3.3 The subcontractor shall have written SOPs for maintaining identification of samples throughout the laboratory.

If the subcontractor assigns unique laboratory identifiers, written SOPs shall include a description of the method used to assign the unique laboratory identifier and cross-reference to the field assigned sample number.

If the subcontractor uses prefixes or suffixes in addition to sample identification numbers, the written SOPs shall include their definitions.

- 3.4 The subcontractor shall have written SOPs describing all storage areas for RFP samples in the laboratory. The SOPs shall include a list of authorized personnel who have access or keys to secure storage areas.
- 3.5 The subcontractor shall have written SOPs describing the method by which the laboratory maintains samples under custody.
- 3.6 The subcontractor shall have written SOPs describing the method by which the laboratory maintains the security of laboratory areas.
- 3.7 The subcontractor shall have written SOPs for tracking the work performed on any particular sample. The tracking SOP shall include:
- A description of the documents used to record sample receipt, sample storage, sample transfers, sample preparations, and sample analyses.
 - A description of the documents used to record calibration and QA/QC laboratory work.
 - Examples of document formats and laboratory documents used in the sample receipt, sample storage, sample transfer, and sample analyses.
 - A narrative step-wise description of how documents are used to track samples.
- 3.8 The subcontractor shall have written SOPs for organization and assembly of all documents relating to each sample delivery group. Documents shall be filed on a SDG specific basis. The procedures shall ensure that all documents including logbook pages, sample tracking records, self-absorption curves, spectra, computer printouts, raw data summaries, correspondence, and any other written documents having reference to the SDG are compiled in one location. The written SOPs shall include:
- A description of the numbering and inventory method, and an example of the document inventory form.
 - A description of the method used by the laboratory to verify consistency and completeness of the case file.

EXHIBIT IV

SPECIFICATION FOR DISKETTE DELIVERABLE

Manual No: 4-21,000-SPEC
Specification No: EMAD-SPEC01, Rev 2
Page No: 1 of 17
Effective Date: 5/3/91

TITLE:

SPECIFICATION FOR PROVIDING THE
ELECTRONIC DELIVERABLE LAB DATA TO
THE ROCKY FLATS ENVIRONMENTAL DATA MANAGEMENT SYSTEM

Approval:

William D. Swan

MANAGER, ANALYSIS AND
MODELING GROUP

5/3/91

DATE

1.0 PURPOSE

This specification describes the responsibilities and activities for providing the electronic deliverable lab data from environmental samples collected at the EG&G Rocky Flats Plant (RFP) site as specified in the scope of work for all contract labs. This data shall be electronically transferred into the Rocky Flats Environmental Database (RFEDS).

2.0 SCOPE

This specification applies to electronic deliverables of analytical data provided by contract labs under the direction of the Environmental Monitoring and Assessment Division (EMAD).

3.0 TERMS/DEFINITIONS

- | | |
|----------------------------|---|
| 3.1 RFEDS | The Rocky Flats Environmental Database. |
| 3.2 Standard RFEDS Format | The format defined in this specification to be used for all RFEDS electronic deliverables. |
| 3.3 EMAD | Environmental Monitoring and Assessment Division. |
| 3.4 ELECTRONIC DELIVERABLE | A PC-DOS (Personal Computer-Disk Operating System) diskette containing ASCII (American National Standard Code for Information Interchange) text files of analytical data in the RFEDS format. |
| 3.5 CAS Number | A unique number assigned to a chemical compound by the Chemical Abstract Service of the American Chemical Society. |

- 3.6 RFEDS SPECIFIC CAS Number A unique number used in RFEDS for specific chemical compounds where there is no formal Cas number.
- 3.7 Test Group Code RFEDS code for the method used to analyze a group of samples.

4.0 RESPONSIBILITIES

- 4.1 The EMAD Analysis and Modeling Group (AMG) is responsible for providing the labs with the RFEDS data format. It is also responsible for quality checking all received electronic deliverables and ensuring compliance with this specification.
- 4.2 The contract labs are responsible for providing electronic deliverables of all analytical data from environmental samples collected under the supervision of EMAD on the RFP site. Contract labs are also responsible for conducting and documenting verification of data and performing regular data backups. The contract labs shall also provide copies of the Chain of Custody (COC) and sample receipt verification forms on a weekly basis.
- 4.3 The EMAD/AMG Analytical Program Chemists are responsible for ensuring that the analytical labs receiving samples are providing electronic deliverables for all results and that any new labs granted contracts are provided with this specification. The Analytical Program Chemists are responsible for ensuring that the requirement to use this specification is incorporated into each laboratory contract statement of work (SOW). The Analytical Program Chemists shall check for data verification procedures during their audits and shall include a statement of data verification procedures in the Audit Report. This shall include data entry quality control and a standard backup procedure.

5.0 PROCEDURE

5.1 Introduction

The overall goal of this activity is to receive good quality, electronic data for direct input into RFEDS. For questions about the electronic deliverable contact Annette Primrose at (303) 966-7041 or Steve Singer at (303) 966-5748. For any questions dealing with contract specifications or items of a chemical nature, contact Karen Schoendaller at (303) 966-5912 or Dr. John Dick at (303) 966-5950.

5.2 Electronic Deliverable

The data shall be provided as an ASCII text file on a PC-DOS disk using the standard RFEDS format. The lab batch identification number (ID) shall be used as the file name. Two copies of each file shall be provided on separate diskettes to Annette Primrose or Steve Singer of the EMAD Analysis and Modeling Group. The diskettes will be clearly labelled with the lab name and the name of the files. The mailing address is:

Annette Primrose/Steve Singer
EG&G Rocky Flats, Inc.
Rocky Flats Plant/EMAD/AMG
Bldg. T130B
P.O. Box 464
Golden, Colorado 80402-0464

5.3 RFEDS Format

The RFEDS format consists of a file containing a header record, a variable number of analytical records and a trailer record. Each record shall be 226 characters long and shall be terminated by a carriage return. The file name shall be derived from the lab batch number. Fields shall be delimited by spaces, not tabs. Data within fields shall be left justified. A complete description and example of the RFEDS format with field names and field lengths is included in Appendix A.

5.3.1 The header record shall consist of the lab project number, the file creation date, and the number of records in the file including the header and trailer records.

5.3.2 Each Analytical record represents one analytical result for a given sample and contains the fields as described in Appendix A. All data shall be left justified within each field, one value per field and fields containing no data shall contain blanks. Each record shall contain the following fields:

FIELD NAME	DESCRIPTION
<u>Sample Number</u>	EG&G sample number. The labs shall not alter the EG&G sample number without prior permission from EMAD/AMG.
<u>Sample Matrix</u>	Matrix type of the sample. Appendix C lists the allowable matrix types.
<u>Lab Batch Number</u>	The number used by the lab to identify a group of samples that were analyzed together.
<u>Lab ID</u>	There is an RFEDS identification code for each contract lab. If a lab has not received one, contact EMAD/AMG.
<u>Lab Blank Sample Number</u>	The lab sample number of the lab blank analyzed with a sample group.
<u>Lab Test Panel Code</u>	The analytical results code based on the analysis type. These are given in Appendix D.
<u>Result Identifier</u>	RFEDS codes that differentiate between actual analytical results and duplicates or spikes. Listed in Appendix E.
<u>CAS Numbers</u>	The formal CAS number assigned to an analyte by the Chemical Abstract Service. If a formal CAS number is not available, use the appropriate RFEDS specific CAS number listed in

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Appendix B. If a CAS number cannot be found, contact Annette Primrose, (303) 966-7041 or Steve Singer, (303) 966-5748. Do not use abbreviations.

Result

The analytical result for a chemical compound. Use numbers, not characters. Use minus signs in front of negative results, and do not use scientific notation. If the result is undetected at the detection limit, put the detection limit in this field and a "U" in the Result Qualifier field.

For Rads, use the actual measured activity even if it is below the detection limit.

Result Qualifier

A code for identifying the reliability of the result. These are listed in Appendix F.

2 Sigma Error

Error data is provided for Radionuclide analyses only.

Unit of Measure

The unit of measure for the result. This shall match the unit of measure used for the contract required detection limit since the CRDL unit of measure is not provided. Appendix G lists the allowable abbreviations.

The unit of measure for blanks and lab control samples shall match the unit of measure used for the sample.

Parameter Name

Name of the compound analyzed. Industry standard abbreviations are acceptable.

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Contract Required
Detection Limit

Use the detection limit specified for the analysis type as required in the contract. For diluted samples, use a detection limit corrected for the dilution factor.

For soils, use a detection limit corrected for percent moisture. For soils that have been diluted, correct for both the dilution factor and percent moisture.

For Rads only, use the actual Minimum Detected Activity of the sample instead of the detection limit.

5.4 Chain of Custody and Sample Receipt Verification Forms

A copy of all COC forms and all sample receipt verification forms shall be provided every Friday for the samples received that week by a contract lab. The copies shall be sent Federal Express or faxed to:

Annette Primrose/Steve Singer
EG&G Rocky Flats, Inc.
Rocky Flats Plant
EMAD/AMG
Bldg. T130B
P.O. Box 464
Golden, Colorado 80402-0464

Fax number: (303) 966-6070

APPENDIX A

Header Record

The first record of each file shall have the Project Number, the file date and the number of records in the file including the header and trailer records. The format is:

Position	Field Length	Field
1 - 20	20	Lab project number or code
21 - 28	8	File Date (MM/DD/YY)
29 - 33	5	Number of records in file

Analytical Records

There shall be one record for each result. All data shall be left justified within the fields and fields with no data must contain blanks. Do not use abbreviations for the CAS number. The format is as follows:

Position	Field Length	Field
1 - 20	20	EG&G sample number
21 - 28	8	Sample collection date (MM/DD/YY)
29 - 33	5	Sample collection time (HH:MM)
33 - 48	15	Lab generated batch number
49 - 56	8	Sample matrix (Appendix C)
57 - 76	20	Lab generated sample number
77 - 81	5	Lab ID - Provided by EG&G
82 - 89	8	Sample preparation date (MM/DD/YY)
90 - 97	8	Analysis date (MM/DD/YY)
98 - 102	5	Analysis time (HH:MM)
103 - 122	20	Lab blank sample number
123 - 132	10	Lab test panel code (Appendix D)
133 - 135	3	Result identifier (Appendix E)
136 - 146	11	CAS number (Appendix B)
147 - 156	10	Result, detection limit if not detected
157 - 161	5	Qualifier (Appendix F)
162 - 171	10	2 sigma error - Rad data only
172 - 179	8	Unit of measure (Appendix G)
180 - 186	7	Retention time-Tentative id compounds only
187 - 216	30	Parameter name
217 - 226	10	Contract Required Detection Limit

Trailer Record

The last record of the file shall consist of three left justified dollar signs.

Position	Field Length	Field
1 - 3	3	\$\$\$

[illegible]

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APPENDIX B

ANALYTE NAME	RFEDS SPECIFIC CAS NUMBER
ACIDITY	10-70-8
ACIDITY, AS CaCO ₃	10-70-8
ADIPATES	10-52-6
ALKALINITY (as CaCO ₃)	10-09-3
ALKYL METHYL ISOQUINOLINIUM CHLORIDE	10-56-0
ARSENATES	10-42-4
ARSENITES	10-43-5
BACTERIA, TOTAL	11-05-2
BETA PARTICLE AND PHOTON RADIOACTIVITY	10-55-9
BICARBONATE AS CaCO ₃	10-13-9
BIOCHEMICAL OXYGEN DEMAND (BOD)	10-26-4
BIOCHEMICAL OXYGEN DEMAND (BOD ₅)	10-26-4
CARBON, TOTAL INORGANIC (TIC)	10-76-4
CARBON, TOTAL VOLATILE ORGANIC	10-25-3
CARBONATE	10-14-0
CBOD ₅	11-03-0
CHEMICAL BIOLOGICAL OXYGEN DEMAND (5-DAY)	11-03-0
CHEMICAL OXYGEN DEMAND (COD)	10-27-5
CHLORINATED BENZENES, N.O.S.	10-16-2
CHLORINATED ETHANE, N.O.S.	10-17-3
CHLORINATED FLUOROCARBONS, N.O.S.	10-18-4
CHLORINATED NAPHTHALENE, N.O.S.	10-19-5
CHLORINATED PHENOL, N.O.S.	10-20-8
CHLORINE, FREE AVAILABLE	11-00-7
CHLORINE, FREE RESIDUAL	11-01-8
CHLORINE, TOTAL RESIDUAL	11-04-1
CHLOROALKYLEETHERS, N.O.S.	10-21-9
COKE OVEN EMISSIONS	10-63-9
COLIFORM, TOTAL	10-46-8
COLOR	10-83-3
CORROSIVITY	10-37-7
CYANIDE, AMENABLE	10-87-7
CYANIDE, FREE	10-71-9
CYANIDES (SOLUBLE SALTS AND COMPLEXES), N.O.S.	10-93-5
DISSOLVED OXYGEN	10-88-8
ENDRIN METABOLITES	10-95-7
ETHYLENEBISDITHIOCARBAMIC ACID, SALTS AND ESTERS	10-96-8
FECAL COLIFORM	10-06-0
FLUORIDE, SOLUBLE	10-72-0
FOAMING AGENTS	10-84-4
GIARDIA LAMBLIA	10-48-0
GLYCOL ETHERS	10-61-7
GROSS ALPHA - DISSOLVED	10-79-7
GROSS ALPHA - SUSPENDED	10-78-6
GROSS BETA - DISSOLVED	10-81-1
GROSS BETA - SUSPENDED	10-80-0
GROSS GAMMA	10-82-2
HALOMETHANE, N.O.S.	10-22-0

ANALYTE NAME	RFEDS SPECIFIC CAS NUMBER
HARDNESS, TOTAL	11-02-9
HARDNESS (as CaCO ₃)	10-92-4
HEXACHLORODIBENZOFURANS	10-65-1
HEXACHLORODIBENZO-p-DIOXINS	10-64-0
IGNITABILITY	10-36-6
LEGIONELLA	10-51-5
meta and para XYLENES	11-57-4
NICOTINE SALTS	10-97-9
NITRATE/NITRITE (as N)	10-28-6
NTA	10-91-3
NVSS	11-06-3
N-ALKYL-N-(2-CYANOETHYL)-1,3-DIAMINOPROPANE	10-58-2
ODOR	10-85-5
OIL AND GREASE	10-30-0
OIL AND GREASE BY IR	11-42-7
ORGANORHODIUM COMPLEX	10-45-7
ORTHOPHOSPHATE	11-36-9
PAH'S	10-53-7
PENTACHLORODIBENZOFURANS	10-67-3
PENTACHLORODIBENZO-p-DIOXINS	10-66-2
PETROLEUM HYDROCARBONS, TOTAL RECOVERABLE	10-90-2
pH	10-29-7
PHENOLS, TOTAL	10-03-7
PHTHALIC ACID ESTERS, NOS	10-23-1
PLUTONIUM-239/240	10-12-8
PNA'S	10-53-7
POLYCYCLIC ORGANIC MATTER (INC. COKE OVEN EMISSIONS)	10-44-6
POLYNUCLEAR AROMATIC HYDROCARBONS	10-53-7
PU-239/240	10-12-8
RADIONUCLIDES	10-62-8
RADIUM-226/228	10-54-8
REACTIVITY	10-38-8
RESIDUE, FILTERABLE	10-33-3
RESIDUE, NONFILTERABLE	10-32-2
RESIDUE, SETTLEABLE	10-74-2
RESIDUE, TOTAL	10-31-1
RESIDUE, VOLATILE	10-73-1
RETORT	10-30-0
SACCHARIN SALTS	10-98-0
SALINITY (FROM CHLORIDE)	10-40-2
SALINITY (FROM SODIUM)	10-41-3
SETTLEABLE SOLIDS	10-74-2
SILICA, DISSOLVED	10-89-9
SOLIDS, NONVOLATILE SUSPENDED	11-06-3
SOLIDS, TOTAL	10-31-1
SOLIDS, TOTAL SETTLEABLE	10-74-2
SOLIDS, TOTAL VOLATILE	10-73-1
SPECIFIC CONDUCTIVITY	10-34-4
STRONTIUM-89/90	11-10-9

ANALYTE NAME	RFEDS SPECIFIC CAS NUMBER
STANDARD PLATE COUNT	10-50-4
STRYCHNINE SALTS	10-99-1
SURFACTANTS (MBAS)	10-75-3
SUSPENDED SOLIDS, NONVOLATILE	11-06-3
TEMPERATURE	10-86-6
TETRACHLORODIBENZOFURANS	10-69-5
TETRACHLORODIBENZO-p-DIOXINS-	10-68-4
TOTAL BACTERIA	11-05-2
TOTAL DISSOLVED SOLIDS (TDS)	10-33-3
TOTAL HARDNESS	11-02-9
TOTAL KJELDAHL NITROGEN	10-07-1
TOTAL ORGANIC CARBON (TOC)	10-35-5
TOTAL ORGANIC HALIDES	10-10-6
TOTAL ORGANIC HALOGEN	10-10-6
TOTAL ORGANIC NITROGEN	10-04-8
TOTAL SOLIDS	10-31-1
TOTAL SUSPENDED SOLIDS (TSS)	10-32-2
TOTAL SUSPENDED SOLIDS (TSS)	10-32-2
TOTAL VOLATILE ORGANIC CARBON	10-25-3
TOTAL VOLATILE SOLIDS (TVS)	10-73-1
TRIHALOMETHANES (THM)	10-77-5
TURBIDITY, NTU	10-08-2
URANIUM, TOTAL	11-09-6
URANIUM-233,-234	11-08-5
URANIUM-233,-238,-239	11-07-4
URANIUM-235/236	10-11-7
VIRUSES	10-49-1
% MOISTURE	10-00-4
% SOLIDS	10-02-6
1,2,3,4,7,8-HEXACHLORODIBENZO-p-DIOXIN	10-15-1
1-(ALKYLAMINO)-3-AMINOPROPANE ACETATE SHAUGHNESSY	10-57-1
1-(ALKYLAMINO)-3-AMINOPROPANE DIACETATE SHAUGHNESSY	10-60-6
1-(ALKYLAMINO)-3-AMINOPROPANE MONOACETATE	10-59-3
2,4-D, SALTS AND ESTERS	10-94-6

APPENDIX C

SAMPLE MATRIX TYPES

WATER	All water-based samples.
SED	All sediment based samples including soil samples.
SLUDGE	Chemical sludge, mixtures of which are neither solid or particularly liquid.
FILTER	Specifically applies to samples composed of the air filters.
CHAR	Material collected from charcoal filters.
FILTSOX	Cloth covers for the charcoal filters.

APPENDIX D

LAB TEST PANEL CODES

VOCCLPTCL	CLP Target Compound List Volatile Organics
SVOCCLPTCL	CLP Target Compound List Semivolatile Organics
PSTCLPTCL	CLP Target Compound List Pesticides/PCBs
TRIPES619	Triazine Pesticides - EPA method 619
CLHERB615	Chlorinated Herbicides - EPA method 615
OCLPEST608	Organochlorine Pesticides/PCBs EPA method 608
SELCOM625	Selected Compounds EPA method 625
SELCOM525	Selected Compounds EPA method 525
SELCO502.2	Selected Compounds EPA method 502.2
PSTPCB508	Pesticides/PCBs EPA method 508
SELCOM505	Selected Compounds EPA method 505
PAHCOM610	PAH Compounds EPA method 610
SELNA607	Selected Nitroso-Amines EPA method 607
DIOX613	Dioxins EPA method 613
DMETCLPTAL	CLP target analyte list Metals-Dissolved
SMETCLPTCL	CLP target analyte list Metals-Total
PDMETCLPTAL	CLP target analyte list Metals-Potentially Dissolved
DMETNOCLP	Non-CLP target analyte list Metals-Dissolved
SMETNOCLP	Non-CLP target analyte list Metals-Total
PDMETNOCLP	Non-CLP target Metals-Potentially Dissolved
WQPL	Water Quality parameter list
DRADS	Dissolved Radionuclides
TRADS	Total Radionuclides

APPENDIX E

RESULT IDENTIFIER

BLK	Blanks including Rad blanks
BS	Method Blank spike.
BSD	Blank spike duplicate.
DUP	Duplicate sample.
MS	Matrix spike.
MSD	Matrix spike duplicate.
MB	Matrix Blank
SPK	Spikes including Rad spikes
TIC	Tentatively identified compounds.
TRG	Regular target compound.
RB	Reagent Blank
REX	Re-extraction
SUR	Surrogate sample.

APPENDIX F

RESULT QUALIFIER

- U A compound was analyzed but not detected.
- J Indicates an estimated value for a tentatively identified compound or analyte that meets the identification criteria but had a result less than the specified detection limit.
- B Compound was found in the blank and in the sample.
- T Compound was found in the TCLP extraction blank and in the sample.
- E Concentration exceed calibration range of the instrument.
- I Indicates interference.
- D Surrogate/matrix spike recoveries were not obtained because the extract had to be diluted for analysis.
- DL Indicates a secondary dilution.
- DF Dilution factor.
- X Result is by calculation.

APPENDIX G

UNITS OF MEASURE

pCi/L	Picocuries per liter
pCi/g	Picocuries per gram
pCi/kg	Picocuries per kilogram
mg/L	Milligrams per liter
ug/L	Micrograms per liter
mg/g	Milligrams per gram
ug/g	Micrograms per gram
mg/Kg	Milligrams per kilogram
ug/Kg	Micrograms per kilogram